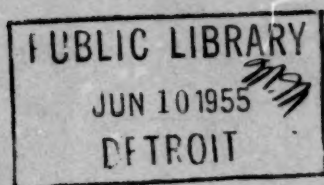PR,

ANALYTICAL
ABSTRACTS

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No. 5, Abstracts 1108-1425

May, 1955

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ANALYTICAL ABSTRACTS

1.—GENERAL ANALYTICAL CHEMISTRY

1108. Measurement and control. (*Instruments and Automation*, 1954, **27** [12, Part 2], 171 pp.).—This number contains among others the following reviews. **Thermometry** (M. F. Behar, 21–35). **Pyrometry** (M. F. Behar, 41–54). **Pressure and vacuum** (M. F. Behar, 57–73). **Instrumentation for chemical analysis and control** (Anon., 109–115). **Determining physical properties and testing finished products** (H. C. Roberts, 118–123).
N. E.

1109. The dualistic and versatile reaction properties of perchloric acid. Illustrative experimental demonstration procedures. G. F. Smith (*Analyst*, 1955, **80**, 16–29).—A historical survey is given of the uses of $HClO_4$ and NH_4ClO_4 and of the manufacture of the acid. The known hydrates of $HClO_4$ are listed and the chemical properties of the acid and its salts are classified. Experiments are described to demonstrate the reactive or non-reactive properties of the acid and its application to particular problems, including the solution of stainless steel for chromium determination and the rapid oxidative destruction of organic matter. The hazards attending the incautious use of $HClO_4$ can be avoided when its properties are fully understood.
A. O. JONES

1110. Perchloric acid and some organic perchlorates. H. Burton and P. F. G. Praill (*Analyst*, 1955, **80**, 4–15).—The history of perchlorates is summarised and methods of preparation of $HClO_4$ are noted. The use of the system acetic acid - perchloric acid as an acetylating agent is discussed, with an indication of the conditions under which its use is not hazardous. The action of $HClO_4$ and some organic perchlorates on various types of organic compounds is described, with particular reference to the formation of alkyl and aralkyl perchlorates. The simple alkyl esters of $HClO_4$ are highly explosive and the possibility of their formation should always be considered (*e.g.*, recovery of alcohol from the filtrates from determinations of K as $KClO_4$ should not be attempted). The outline given of the fundamental chemistry of $HClO_4$ should serve for the consideration of the hazards that may occur in its use. Each expt. should be considered separately as there are no general rules as to when or when not its use is dangerous. (149 references.)
A. O. JONES

1111. Application of Versene to separations with 8-hydroxyquinoline. R. P. Taylor (*Dissert. Abstr.*, 1954, **14** [10], 1522).—Versene (ethylenediamine-tetra-acetic acid) and versenate complexing is studied as a method of improving the selectivity of extraction of metal oxines from $CHCl_3$ soln. The metals studied are divisible into three groups according to their extraction behaviour: Mo, Ti, W and U are 95 per cent. extracted at pH < 8; Al, Co, Fe^{III}, Mn and Ni are extracted only at pH values

> 8·1; and Cu and V are less than 95 per cent. extracted at pH < 8, but this yield can be improved by using Ca versenate instead of Versene; reasons for this are given. The preparation of the $CHCl_3$ solutions by complete extraction from standard aq. solutions containing metal ions is described and their absorbancies are measured. A theoretical equation is derived from the various equilibria involved. Theoretical extraction curves are compared with experimental curves. A conductimetric titration method involving a constant-current conductance cell is also studied. The cell consists of a 150-ml beaker into which two platinum primary electrodes and two tungsten probe electrodes have been fused. A constant current is passed through the primary electrodes and the voltage drop is measured across the probe electrodes. The conductance is inversely proportional to this voltage drop. This method is applied to the determination of Mo and W 8-quinolinoxides, and to a number of classical conductance titrations, and also to the non-aqueous titration of perchloric acid with aniline. A second type of conductance cell is proposed in which calomel electrodes are used instead of W for the secondary, or probe, electrodes.
S.C.I. ABSTR.

1112. Purification of *cyclo*hexane for ultra-violet spectroscopy. R. H. Linnell and S. Umar (*Chemist Analyst*, 1954, **43** [4], 90–91).—A refluxing apparatus comprises a reaction vessel between a flask and a condenser; the vessel is made by evacuating and sealing off the jacket of a condenser and filling the inner tube with glass beads. Ground-glass joints may be sealed with a "Halocarbon grease." *Procedure*—900 ml of technical-grade *cyclo*hexane are refluxed, while 200 ml of fuming sulphuric acid are dropped into the top of the condenser at such a rate that addition is complete in 6 hr. The *cyclo*hexane is separated from the acid, washed with dil. NaOH solution, then with H_2O, and distilled from an all-glass column with an efficiency of $\ll 5$ theoretical plates. At least 90 per cent. of the *cyclo*hexane is recovered; the absorbancy at 230 mμ was reduced from 1·60 to 0·07.
H. P. PAGET

1113. Investigation of analytical chemistry on filter-paper. VIII–X. Analysis of the inorganic anions by paper chromatography. III–V. III. Influence of 27 cations on Cl′. S. Nakano (*J. Chem. Soc. Japan*, 1954, **75** [2], 150–152).—Paperchromatographic absorption of 27 chlorides (2 μl of 100 μg per 10 μl of soln.) was studied at 25° C with a solvent (80 per cent. ethanol), the partition velocity being 5 hr. per 20 cm. The R_F values of cations and chloride ions were examined closely. Characteristic R_F values for Cl′ are 0·65 to 0·72, but they are affected by the cation. Within the same sub-group of the periodic table, the larger the at. wt., the smaller the R_F of Cl′. This result may be due to the fact that the solubility of a chloride in ethanol decreases with the at. wt. in the same sub-group. **IV. Influence of 25 cations on NO₃′** (*Ibid.*, 1954, **75** [2], 152–154).—A similar

expt. was carried out with nitrates. The same relation between the R_F value and the solubility of the nitrate in ethanol was found to hold. R_F values for NO_3' are $\simeq 0.75$, but R_F values for cations are different from those of chlorides. **V. Influence of 25 cations on SO_4''** (*Ibid.*, 1954, **75** [2], 154–156). —A similar experiment was carried out with sulphates. The relation which was found in the previous examples is again true. K. SAITO

1114. Investigation of analytical chemistry on filter-paper. XI. The behaviour and the separation of sulphate and fluorosilicate by paper chromatography. S. Nakano (*J. Chem. Soc. Japan*, 1954, **75** [3], 328–330).—The paper-chromatographic distribution of aq. soln. (100 μg per 0·01 ml) of SO_4'' and SiF_6'' is studied in aq. NH_3 soln., methanol and methanol-28 per cent. aq. ammonia mixture $(4 + 1)$. In aq. ammonia (2 to 15 N), R_F values for both anions are $\simeq 0.9$ and the influence of cations (except Li') is small. In methanol, the Na salt of SiF_6'' remains stationary and is separated from SO_4'' ($R_F \simeq 0.6$), whilst the K and Li salts and fluorosilicic acid have R_F values approx. 0·8. For analytical procedures the Na salt seems best. The relation between the R_F value and the solubility, which was reported in the previous paper (*J. Chem. Soc. Japan*, 1954, **75** [2], 154–156), holds for these solvents as well. **XII. Separations of small amounts of Ag and Hg (I) from large amounts of Cu, of small amounts of Ag from large amounts of Hg (I) and of the silver group from other groups by paper chromatography.** S. Nakano and S. Shimada (*Ibid.*, 1954, **75** [5], 506–509).—For the separation of Hg^I and Ag from other elements, 0·2 N HCl is the best solvent (limit of detection, 0·1 μg of Ag or 0·2 μg of Hg per 0·002 ml); both of them stay at the initial point on the paper. This spot is oxidised by keeping the paper within a vessel containing a small amount of bromine, and then submitted to ascending paper chromatography once again, using 0·2 N HCl as solvent, to separate Ag and Hg (limit of detection of Ag in Hg, 0·5 μg of Ag; Hg:Ag = 800:1). Ag can also be detected in a soln. containing $Cu^{..}$ in 0·2 N HCl (limit of detection, 0·4 μg of Ag per 0·002 ml; Cu:Ag = 2000:1). When Hg is present, the spot is treated as mentioned above and both Hg and Ag can be detected (limit, 0·5 μg of Ag, 0·4 μg of Hg per 0·002 ml; Ag:Ag = 1600:1; Cu:Hg = 2000:1). **XIII. Influence of large amounts of ferric ion on the R_F values of other elements and the detection of small amounts of metals in iron ores by paper chromatography.** S. Nakano and S. Shimada (*Ibid.*, 1954, **75** [5], 509–512).—The most favourable solvent contains 74 per cent. (by vol.) of *iso*pentanol and 26 per cent. of conc. HCl. The influence of large amounts (2 to 50 times) of Fe appears different from ion to ion, according to their R_F values. Ions of R_F values < 0·20 (Na, Ca, La, Mn, Ni, etc.) are not affected by < 50 times the concn. of $Fe^{...}$. Ions of R_F values 0·2 to 0·6 (Cu, Ag, Be, Ti, Bi, $UO_2^{..}$, WO_4'', Co, etc.) are affected by > 5 times the concn. of Fe. Ions of R_F values 0·60 to 0·90 (Zn, Cd, Sn, As, Sb, MoO_4'', etc.) form bands very close to the Fe band. By means of this solvent, the detection of minor constituents in pyrites is effected. With the aid of a variety of org. reagents, the main impurities can be detected in a soln. of the ore in aqua regia. K. SAITO

1115. Organic co-precipitants (collectors). I. Theoretical foundations of the action of organic co-precipitants. V. I. Kuznetsov (*Zh. Anal. Khim.*, *SSSR*, 1954, **9** [4], 199–207).—The principles to be followed in selecting organic co-precipitants are (*i*) use of a compound containing a relatively large organic part, and the formation of a compound of the ion to be precipitated with structural similarity to the co-precipitant, *e.g.*, the reaction of methyl violet with $Zn(CNS)_4''$, which has been used for the precipitation of Zn from dilute solution, can be made more effective by the addition of excess of NH_4CNS to precipitate methyl violet thiocyanate, with which traces of zinc left in solution are co-precipitated, and (*ii*) use of an indifferent co-precipitant, a non-salt-forming organic compound, the action of which can be considered as extraction with solid solvents. Examples of (*ii*), *e.g.*, the use of dialkyl ethers of dimethylglyoxime for co-precipitation of the nickel compound of dimethylglyoxime, have to be sought empirically. G. S. SMITH

1116. Systematic analysis of anions by micro- and semi-micro techniques. F. de Leo, R. Indevina and A. Bellino (*Ann. Chim., Roma*, 1954, **44** [11], 859–874).—Full experimental details of a systematic (qualitative) micro-analytical method for anions are described; it is based on an initial separation into sol. and insol. zinc salts. Confirmatory tests are also given. C. A. FINCH

1117. Quantitative analysis without separation. V. Gravimetric analysis without separation for the systems, Pb - Ba and Ce - Th. N. Unohara (*J. Chem. Soc. Japan*, 1954, **75** [3], 287–289).—Thermal decomp. curves are studied with the mixed ppt. of lead and barium oxalates and with cerium and thorium oxalates. In mixed ppt., the decomp. of oxalates takes place at a lower temp. than in a pure state. Quantitative analyses of these mixtures can be carried out only when the amounts of both compounds are approx. equal. **VI. Gravimetric analysis without separation for the systems, Mg - Na, Mg - K and Mg - Na - K** (*Ibid.*, 1954, **75** [3], 289–292).—In the mixed ppt. with alkaline-earth sulphates, $MgSO_4$ is decomp. at a little lower temp. than in the pure state, but the decomp. of $MgSO_4$ is retarded by the presence of K or Na sulphate, possibly owing to the low melting-point of alkali sulphates. Quant. analysis of Mg without separation is possible when the amount of Na or K present is not more than twice that of Mg. **VII. Gravimetric analysis without separation for the systems, Cu - Mn and Cu - Cd** (*Ibid.*, 1954, **75** [4], 423–425).—The thermal decomp. of mixed ppt., *e.g.*, $CuSO_4$ plus $MnSO_4$; and $CuSO_4$ plus $CdSO_4$, is studied. The decomp. of $CuSO_4$ does not end until the decomp. of $MnSO_4$ begins, making the analysis without separation difficult. In the Cu - Cd system, $CdSO_4$ does not start to decomp. until the decomp. of $CuSO_4$ ends. The amount of copper can be found from the difference in weight, when the amount of $CdSO_4$ is < 20 times the weight of $CuSO_4$. **Gravimetric analysis without separation for the systems, Zn - Cd and Zn - Pb** (*Ibid.*, 1954, **75** [5], 544–545).—The thermal decomp. of $ZnSO_4$, $CdSO_4$, $PbSO_4$, $ZnSO_4$ plus $CdSO_4$, and $ZnSO_4$ plus $PbSO_4$ are studied with a thermobalance, and the results compared with those obtained by other workers. The determination of the amount of Zn from the decrease of weight in the heating of $ZnSO_4$ - $CdSO_4$ is not satisfactory, nor is the determination of Zn in $PbSO_4$ - $ZnSO_4$. K. SAITO

1118. Use of redox indicators in acidimetric titrations. E. Rancke-Madsen, H. Skarbye-Nielsen and K. Østergaard (*Acta Chem. Scand.*, 1954, **8**

[8], 1414–1420).—Weak bases ($pK_B < 10$) such as acetate ion, succinate ion and pyridine can be titrated acidimetrically with reasonable accuracy by the use of the redox system ceric - cerous and nitroferroin or ferroin. The weak acid was titrated past the equivalence point to colour transition of the indicator system, and then the over-titration was corrected by means of a blank experiment carried out on pure water, so that the end volume in the titration of the weak base was almost identical with that in the titration of water.   N. E.

1119. Catalysis and induction in redox titrations. VI. Catalysis.

A. Schleicher (Z. anal. Chem., 1955, **144** [2], 100–105).—In the titration of $Fe^{..}$ by $KMnO_4$ in HCl, the evolution of chlorine can be reduced by inhibition through self-induction. In the opposing effect of a large amount of added Mn^{II}, the newly formed Mn^{III} acts as a catalyst. In the mechanism proposed, this easily reducible Mn^{III} performs the functions originally assigned to hypothetical unstable Fe with a valency higher than 3. The added Mn^{II} reduces the Mn^{VII} thus:— $Mn^{.......} + 5 Mn^{..} = Mn^{..} + 5 Mn^{...}$ (newly formed) and the newly formed $Mn^{...}$ oxidises Fe^{II} according to $5 Fe^{..} + 5 Mn^{...} = 5 Fe^{...} + 5 Mn^{..}$ (regenerating Mn^{II}). Induction and catalysis differ in degree rather than in kind; changes in electrical field strength in solution, measurable by changes in potential, occur by induction processes analogous to those in electrical solenoids. This is similar to the reduction of permanganates, chromates and vanadates by inner electrolysis with Zn.

D. R. GLASSON

1120. High-frequency titration.

K. Cruse and R. Huber (Angew. Chem., 1954, **66** [12], 625–633). —The fundamentals of high-frequency titration and, specifically, the properties of analysers "without electrode contact," are discussed, and the types of equipment used in practice are classified. Measurements with a heterodyne apparatus are described; the construction of the apparatus for use in high-frequency titration is also given. The importance of the optimum concentration range and of the use of the apparatus for the measurement of the dielectric constant and the conductivity is described. The applications of this technique in various types of titration are outlined. (51 references.)

C. A. FINCH

1121. Coulometric determinations at constant current in unstirred solutions.

L. Gierst and P. Mechelynck (Anal. Chim. Acta, 1955, **12** [1], 79–91). —A constant-current coulometric method is described in which the "transition time" only is measured in an unstirred soln. with a stationary electrode. The transition time is the interval between the establishment of the capacity charge on the electrode and the increase in potential that occurs when the concn. of the substance at the surface of the electrode falls to zero. Conditions are designed to minimise effects other than that of the diffusion current. The subject is treated mathematically and two "transitometers" are described for providing the necessary electrical and timing devices. Determinations occupy less than 1 min. and the accuracy is $\simeq 0.2$ per cent.

W. C. JOHNSON

1122. Theory of adsorption chromatography for liquid mixtures.

G. G. Baylé and A. Klinkenberg (Rec. Trav. Chim. Pays-Bas, 1954, **73** [11], 1037–1057).—The theory of adsorption chromatography for liquid multi-component mixtures is critically reviewed (21 references). The shape of a chromatogram in development chromatography depends on the mathematical characteristics of the adsorption isotherm governing the process. The subject is treated mathematically.   S.C.I. ABSTR.

1123. Gradient and rate aspects in paper chromatography.

B.-T. Ackerman and H. G. Cassidy (Anal. Chem., 1954, **26** [12], 1874–1876).—Properties of ten types of filter-paper used for upward-flow chromatography are listed, viz., average thickness, ratio of weight of wet paper to dry paper, rate of change of thickness of film of water with varying distance from the reservoir and rate of water rise. The role of these properties, hydrogen bonding and molecular size within homologous series are used to interpret the behaviour of R_F values of solutes.

D. A. PANTONY

1124. Use of "carboxyl" paper in paper chromatography.

U. Ströle (Z. anal. Chem., 1955, **144** [4], 256–264).—The chromatographic separation by ion exchange of basic substances on paper containing hydroxycellulose rich in carboxyl groups has been examined. The basic amino acids have been used as examples. The theory of the process is discussed. By variation of the carboxyl content and therefore of the ion-exchanging capacity of the paper, and by correct adjustment of the pH value, the buffer capacity and the cationic strength of the developing solution, it is possible to effect a satisfactory separation of arginine, lysine and histidine.

A. J. MEE

1125. Flowing chromatography on a circular paper pack.

K. V. Giri and D. B. Parihar (Nature, 1955, **175**, 304–305).—By the technique described, components of a mixture are separated by circular paper chromatography on a paper pack and then eluted with a device which facilitates the flow of solvent from the edges of the paper pack. The apparatus is illustrated. Applications include separations into components of mixtures, e.g., of rhamnose and maltose (by n-butanol - ethanol - water), of rhamnose, glucose and maltose (by butanol - acetic acid - water), and of methyl orange, neutral red and methylene blue (by 80 per cent. ethanol).   C. H. WHITTON

1126. Electro-osmosis in paper electrochromatography with electrodes on the paper.

S. E. Wood and H. H. Strain (Anal. Chem., 1954, **26** [12], 1869–1872).—Marked filter-paper sheets ($\simeq$ 70-cm long, with variable width) that have been washed with M HNO_3 are soaked in 0.1 M lactic acid and the excess of acid is removed; they are then treated with the indicators: H_2O_2, formaldehyde, thioacetamide, allylthiourea and quinol (0.1 M in 0.1 M lactic acid, $50\mu l$), and 3.4 per cent. radio-sucrose and radio-dextran at the marked points. The paper is clamped between polythene sheets with platinum electrodes placed at variable intervals on the paper. The voltage is adjusted to 10 V per cm. Spraying with locating soln. of 6 N NaOH and 0.1 M $AgNO_3$ in 8 per cent. aq. NH_3 (1 + 1), or radio-counting, is used to display the indicator movements. R_F values without applied potential are virtually unity, save that for quinol, and, within experimental error, migrations of the indicators are identical, so that electro-osmosis is the controlling factor in the very small migrations. Details of movements of the indicators are tabulated, and from the slight variation of migrations with varying positions of the indicator with respect to the electrodes, with wetness of the paper and with time of electrolysis,

it is concluded that complex resistive forces are in operation, resulting from changes in distribution in the supporting electrolyte.   D. A. PANTONY

1127. Ion-exchange resins in analytical chemistry. R. Pallaud (*Chim. Anal.*, 1955, **37** [1], 16–19).— A review is presented that deals with the classification of ion-exchange resins and their physicochemical characteristics. The importance of the extent of the acidic or basic character of the exchanger resins in deciding which will be effective in a particular case is emphasised.   A. J. MEE

1128. Some experimental evidence of collision processes in spectrochemical analysis. L. W. Strock (*Applied Spectroscopy*, 1954, **8** [3], 105–114). —With the author's germanium-metal dilution method (*Applied Spectroscopy*, 1953, **7**, 64), calibration has been effected by using a series of synthetic silicate melts of $CaSiO_3$ and $CaMg(SiO_3)_2$ and twenty intermediate mixtures. It is concluded, from a study of the slopes of the calibration curves of Ca, Mg and Si, that there is energy interaction between the atoms of Mg and Si. This results in an enhancement of silicon intensity and relative weakening of magnesium intensity as the population of magnesium atoms in the arc increases.
    B. S. COOPER

1129. The preparation of nitrogen samples for mass-spectrographic analyses. P. F. Holt and B. P. Hughes (*J. Chem. Soc.*, 1955, 95–97).—A modified Dumas procedure is described for preparing nitrogen samples from isotopically labelled substances for mass-spectrographic analysis. Incomplete combustion is avoided by using a high temp. of combustion (1050° C) and a nickel oxide furnace filling. The correction for air contamination should be calculated from the argon-40 peak rather than the oxygen-32 peak.   C. H. WHITTON

2.—INORGANIC ANALYSIS

1130. Development of a new scheme for the qualitative analysis of cations without the use of hydrogen sulphide. E. L. Serbinovskaya (*Tr. Mosk. Tekhn. In-ta Ryšbnoi Prom-sti i Rhozyaĭstva im. Mikoyana*, 1953, [5], 165–169; *Referativnyĭ Zh., Khim.*, 1954, Abstr. No. 29,293).—After separation of the silver sub-group cations, Hg, Bi, Cd, Al, Cr, Mn, Sb, Sn and Fe are pptd. by excess of $(NH_4)_2CO_3$; the filtrate contains Cu, Zn, Ni, Co and As^V.
    E. HAYES

1131. Inorganic analyses involving the joint use of the ion-exchange column and the polarograph. L. Gierst and L. Dubru (*Bull. Soc. Chim. Belg.*, 1954, **63** [8–10], 379–392).—Some analytical potentialities of combining ion-exchange and polarographic methods are briefly discussed and illustrated by reference to the determination of traces of Zn in Cd. A preliminary separation is carried out on Dowex-50 resin with 0·25 M ammonium citrate, at pH 4·0, as the eluting agent. The limiting sensitivity of the method is about 0·002 per cent. of Zn in Cd; 0·1 per cent. of Zn can be determined with an accuracy of ± 2 per cent.   A. JOBLING

1132. Application of ion exchangers in analytical chemistry. I. Separation of lithium-, sodium- and potassium-ions. D. Jentzsch and I. Frotscher (*Z. anal. Chem.*, 1955, **144** [1], 1–8).—Wofatit KPS 200 affords quant. separation of Li·, Na· and K· consecutively. Mixtures containing about 20 milli-

equiv. of the 3 elements in approx. 1 : 1 : 1 ratios are separable on columns 110 cm long with 0·1 N HCl as eluting solution. Wofatit F quant. separates Li and Na only under special conditions. Selectivity is increased by application of vacuum to the exchange column, permitting use of Wofatit of particle size < 0·15 mm.   D. R. GLASSON

1133. Application of ion exchangers in analytical chemistry. II. Adsorption of elements on anion exchangers in hydrochloric acid solution. D. Jentzsch and I. Frotscher (*Z. anal. Chem.*, 1955, **144** [1], 17–25).—The adsorption of Al, As, Pb, Cd, Ca, Fe, In, Cu, Co, K, Li, Mg, Mn, Na, Ni, Sn and Zn on Wofatit L150 is studied. Possible separations arising from differential adsorption at various concn. of HCl (0·005 N to 12·5 N) are investigated. Advantages over earlier chromatographic methods are emphasised. Determination of elution constants and further studies of the metal-ion complexes in acid or salt solutions are contemplated to establish more precise conditions of separation.   D. R. GLASSON

1134. Studies on hygroscopicities of precipitates with the use of a hygro-balance. III–IV. H. Amano (*J. Chem. Soc. Japan*, 1954, **75** [5], 499–506).—**III.** **Relative hygroscopicities of the precipitates of Mn, Zn, Hg, Pb, Se, Te, Ti and Si and of several standard substances.** The hygroscopic nature of various forms in which the above elements are weighed is studied with the aid of a hygro-balance, and the differences are discussed. TiO_2 and SiO_2 absorb water to a marked extent when they have been ignited at 700° C but do not when ignited at 1000° C. **VI.** **Relative hygroscopicities of the precipitates of K, Na, Mg, Sr, Mo, V, W and Ag and of several standard substances.** Among a variety of forms in which the above elements are weighed, $K_2Na[Co(NO_2)_6]$, NaCl, Na_2SO_4, $MgCO_3$, MgO, $MgSO_4$, $MgNH_4PO_4.6H_2O$ and $MgNH_4PO_4.H_2O$ show marked hygroscopicities.   K. SAITO

1135. Titrations in strongly alkaline media. VIII. Titration of hydrogen peroxide with potassium ferricyanide. J. Vulterin and J. Zýka (*Chem. Listy*, 1954, **48** [4], 619–620).—The method is based on the oxidation of H_2O_2 to O with $K_3Fe(CN)_6$ in strongly alkaline soln.; the reaction taking place is expressed by $2K_3Fe(CN)_6 + 2KOH + H_2O_2 \rightarrow 2K_4Fe(CN)_6 + 2H_2O + O_2$.   Procedure—Dilute 0·5 ml of an approx. 2 per cent. soln. of H_2O_2 to 50 ml with 30 per cent. KOH and titrate potentiometrically with 0·1 N $K_3Fe(CN)_6$. The end-point is recognised by a potential drop of approx. 400 mV; the potential of inflexion is + 50 mV.
    G. GLASER

1136. Lithium isotope determination by neutron activation. L. Kaplan and K. E. Wilzbach (*Anal. Chem.*, 1954, **26** [11], 1797–1798).—Samples of Li_2SO_4 containing ^6Li and ^7Li, including one of known isotopic ratio, are dissolved in a known vol. of water, and 0·5-ml aliquots are weighed into quartz ampoules. These, and blanks of water and 0·1 N H_2SO_4, are subjected to a flux of 10^{12} neutrons per sq. cm per sec. for 2 weeks. The ampoules are broken under vacuum and the issuing gases, except tritium, are condensed in liquid nitrogen; the gaseous tritium and that obtained by the action of Zn on the blank solutions are analysed by a standard method (*Anal. Abstr.*, 1954, **1**, 2040). Precision is claimed to be ± 1 per cent. for ^6Li.
    D. A. PANTONY

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1137. Amperometric detection of ions [of lithium, sodium and potassium] in paper chromatograms. G. de Vries and E. van Dalen (*Rec. Trav. Chim. Pays-Bas*, 1954, **73** [11], 1028–1032).—The dried chromatogram in which chlorides or perchlorates of Li, Na and K have been chromatographed with a solvent mixture of 3 pt. of pentanol and 7 pt. of methanol is passed between conducting rollers driven by a synchronous motor, which gives an audible signal every 3 sec. and takes 10 min. to scan a 15-cm paper. A fixed potential of 80 to 200 V is applied to the rollers and the current passing is read on a spot galvanometer every 3 sec. The lower voltage is used for the results recorded since increase in voltage, although it increases the height of the max., also increases the background, and increase in dampness of the atmosphere has the same effect. The sensitivity may be increased by putting several spots side by side on the start line. By this method $< 1 \mu g$ of Li, $< 3 \mu g$ of Na and $< 5 \mu g$ of K can be detected in the presence of 100 μg of any other of these three ions. The sensitivity with perchlorate is less and no separation is attained with sulphates either with this solvent, or with 80 per cent. or 100 per cent. methanol.

E. J. H. BIRCH

1138. Mixed indicator for soda-ash titrations. M. Greene (*Chemist Analyst*, 1954, **43** [4], 91).— The colour of methyl red at the end-point of a carbonate titration is difficult to match in a buffer; the following indicator mixture gives an easily discernible transition from green to yellow. Solutions (0·1 per cent.) of bromocresol green, thymol blue and cresol red are mixed in the proportions 14 to 6 to 1, and 6 to 8 drops of the mixture are added per 100 ml of carbonate solution. The endpoint may be matched with a solution of 15·3 g of potassium dihydrogen phosphate in 100 ml of H_2O.

H. P. PAGET

1139. Investigations on the determination of potassium in solution by the Geiger - Müller counter. H. Dresia (*Z. anal. Chem.*, 1955, **144** [2], 81–90).— The counter and test solutions are placed in concentric tubes. Chemical composition, *e.g.*, different anions, has no influence on the measurements, provided the electron densities are not appreciably different. The dependence on the solution density is quant. expressed by an exponential absorption function determined from single standard measurements with pure substances. Possible analytical errors caused by other radioactive substances such as Th and U are discussed and evaluated by standard measurements with UX_2; the K determination is affected only by amounts of Th and U about 1000 times the concn. usually present in natural ores. A max. counter efficiency of 14 per cent. is obtained with a 1-mm space between the tubes, permitting determination of K to an accuracy of 2 to 3 per cent. at the lower concn. limit of 20 mg per ml.

D. R. GLASSON

1140. Argentimetry of potassium and organic N-containing bases with sodium tetraphenylboron. W. Rüdorff and H. Zannier (*Angew. Chem.*, 1954, **66** [20], 638–639).—The argentimetric determination of K with $NaB[C_6H_5]_4$ is improved when K is precipitated with excess of $NaB[C_6H_5]_4$, filtered, and BC_6H_5' is determined in acetone by Volhard's argentimetric method. The K is pptd. by addition of the reagent (2 per cent. solution) to an aq. soln. of acidity not more than 0·1 N, with the addition of a few ml of 0·2 N aluminium nitrate solution

to prevent increase of acidity and to effect coagulation of the ppt. The ppt. is filtered and washed with saturated $KB[C_6H_5]_4$ solution and finally water. The ppt. is dissolved in 20 to 30 ml of acetone, excess of 0·5 N $AgNO_3$ is added and the whole is diluted with water to 100 ml. The mixture is shaken with ether (to dissolve the $AgB[C_6H_5]_4$ ppt.) and ferric ammonium sulphate is added, and excess of Ag is titrated with 0·05 N NH_4CNS. A number of amino-acid cations do not interfere, but errors arise in the presence of $NH_4{\cdot}$ and Rb and Cs salts. Certain organic bases and alkaloids are pptd. by the reagent and the determination of amidopyrine, atropine, pyridine, phenazone, benzidine and hexamine is possible by this method. C. A. FINCH

1141. Determination by radioactivation of small amounts of rubidium and caesium in sea-water and related materials of geological interest. A. A. Smales and L. Salmon (*Analyst*, 1955, **80**, 37–50).—Neutron-radioactivation analysis has been applied to the determination of Rb and Cs in the range 10^{-5} to 10^{-8} g. Rb in the irradiated sample with additional Rb_2SO_4 as carrier is first separated as $RbClO_4$, which is dissolved in HCl. The Rb is then pptd. as cobaltinitrite, this is dissolved in dil. HNO_3 and the Rb is finally pptd. as the chloroplatinate. This is weighed to ascertain the chemical yield and the activity is determined with a suitable counter. An irradiated standard is treated similarly. Cs is pptd. first as the double salt with bismuth iodide and finally weighed and counted as the chloroplatinate. The method was used for the determination of Rb and Cs in sea-water, after a preliminary concn. on a cation-exchange resin, and directly in seaweeds, marine sediments and coals. The Rb and Cs contents of North Atlantic sea-water were found to be 120 and 0·5 μg per litre, respectively. Determination of mg amounts of Na by neutron radioactivation is mentioned, chemical separation being avoided by the use of γ-ray spectrometry. A. O. JONES

1142. Detection of gold in the presence of palladium, platinum and other metals. J. Doležal and P. Beran (*Chem. Listy*, 1954, **48** [4], 618–619).—A new reagent for the detection of $Au{\cdots}$ ions by the usual procedure of reducing these to a blue-violet or red-violet ppt. of metallic Au, is prepared by dissolving ascorbic acid (8·85 g), complexone III (0·1 g) and formic acid (4 ml) in 1 litre of water. Interference by Pd and Pt can be eliminated by adding 0·1 M complexone III before the application of the reagent. G. GLASER

1143. Ion-exchange method for determination of alkali metals in presence of calcium and magnesium. O. Samuelson and E. Sjöström (*Anal. Chem.*, 1954, **26** [12], 1908–1910).—When soln. of $K{\cdot}$ with $Ca{\cdot\cdot}$ (as chlorides) in 60 per cent. ethanol are stirred (30 min.) with an anion-exchange resin [Dowex 2 (100 ml) treated with 0·2 M disodium ethylenediaminetetra-acetate (500 ml) and washed with water (1500 ml)] (**I**) and the slurry is made up into a normal exchange column from which the effluent is passed through the same exchanger in free base form [Dowex 2 (100 ml) treated with N NaOH (1000 ml) and washed with water] (**II**), separation is quant. after elution with 60 per cent. ethanol (80 ml). The liberated KOH is titrated with standard acid. The presence of $Mg{\cdot\cdot}$ prevents full recovery of the $K{\cdot}$. A similar procedure with a mixture of equal volumes of **I** and **II** allows the complete separation of $K{\cdot}$, $Na{\cdot}$ and $Li{\cdot}$ from $Ca{\cdot\cdot}$

and Mg··, provided that 100, 400 and 1000 ml, respectively, of the 60 per cent. aq. ethanol wash-liquid are used. For K determinations, a relative error of 0·2 per cent. is claimed. To reduce the time of Na determinations a third similar method, in which equal volumes of **I** and the acetate form of the resin (Dowex 2 treated with 1·5 M Na acetate to remove Cl′ and then washed with water until neutral to methyl red) are used, is recommended; in this procedure, relative error is given as 0·3 per cent. Solutions made slightly acid with HCl or H_3PO_4 can still be treated by this method.

D. A. PANTONY

1144. The use of ion-exchange resins in analytical chemistry. XXVIII. The determination of the alkali metals in the presence of calcium, magnesium and other metals. O. Samuelson, E. Sjöström and S. Forsblom (*Z. anal. Chem.*, 1955, **144** [5], 323–328).—A method for estimating the alkali metals in solutions containing Ca··, Mg··, V·····, Fe···, Al···, Cu··, Ni··, Co··, Mn·· and Zn·· is described. *Procedure*—The solution is passed through two columns containing Dowex 2, a strong anion-exchange resin. The first column contains two layers, the upper one being a mixture of equal parts of resin in the acetate form and in the ethylenediaminetetra-acetate form and the lower one resin in the oxalate form. The second column contains the hydroxyl form. The alkali-metal hydroxides, which are eluted from the second column, are titrated with standard acid. The maximum error in the determination of K and Na is 0·6 per cent. Small amounts of acid in the original solution do not interfere. (See preceding abstract.) P. S. STROSS

1145. Rapid colorimetric determination of copper by means of 2-isatoxime methyl ether in the presence of other metals. L. Diviš and J. Škoda (*Chem. Listy*, 1954, **48** [4], 539–541).—A highly selective method for the colorimetric determination of Cu is described, based on the formation of a coloured chloroform-soluble complex by the reaction between Cu·· and 2-isatoxime [an isatin oxime] methyl ether (**I**). By working in weakly acidic conditions (pH 5 to 7), and by adding Na K tartrate, Cu^{II} can be determined in the presence of a 2000-fold excess of Ag·, Zn··, Cd··, Pb··, Mn··, Fe···, Co·· or Ni··; interference by Hg·· can be eliminated by the masking action of KI. *Procedure*—Treat the sample containing 1 to 20 μg of Cu^{II} and other metals at pH 5 to 7 with 30 per cent. aq. Na K tartrate (4 ml), 0·05 per cent. aq. **I** (2 ml) and, if Hg·· is present, 30 per cent. aq. KI (1 ml). Dilute the soln. to 10 ml, shake for 2 min. with chloroform (5 ml) and measure the absorption using a green filter (min. absorption at 540 mμ). G. GLASER

1146. Compleximetric determination of copper in acid and ammoniacal solution using Chromazurol S as indicator. M. Theis (*Z. anal. Chem.*, 1955, **144** [4], 275–277).—Copper gives a coloured lake with Chromazurol S (Geigy); in solutions of pH 6 to 6·5 it is blue, and in solutions of pH 8 to 10 it is reddish-violet. On the addition of an equivalent amount of disodium ethylenediaminetetra-acetate solution (**I**), the colour changes to light green. Chromazurol S can thus be used as an indicator in the titration of Cu with **I**. The determination in weakly acid solution is unaffected by alkaline earths, but Al, Zn, Cd, Pb, Co and Fe^{III} interfere. Copper cannot be determined by this method in the presence of Cd, Co and Zn. The interference from Pb cannot be removed by the addition of tartrate ion, but

Fe^{III} and Al can be masked by NaF. In ammoniacal solutions, Zn, Co, Cd, Ni, Pb and the alkaline earths interfere. A. J. MEE

1147. The universal application of the photometric determination of copper in metal analysis by extraction of the diethyldithiocarbamate complex. H. Pohl (*Anal. Chim. Acta*, 1955, **12** [1], 54–63).—The influence of Bi, Ni, Co and Fe on the diethyl-dithiocarbamate method for Cu determination has been investigated. Extinction curves in the range 200 to 600 mμ are produced for these five metals, the complexes being extracted with $CHCl_3$ from solutions containing citric acid, NH_4Cl and an excess of aq. NH_3. At 436 mμ, the max. used for determination of Cu, the other four metals show absorption, and the magnitude of their respective absorptions is measured in terms of Cu. The Bi complex is not extracted by $CHCl_3$, if Pb is present; as Bi is generally found only in lead alloys its effect is eliminated. Nickel can be eliminated by the previous addition of dimethylglyoxime and extraction of the complex with $CHCl_3$, and the same reagent prevents interference from Co (*Brit. Abstr. C*, 1950, 265). Fe, Co and Ni can be sequestered with ethylenediaminetetra-acetic acid but the concn. of diethyldithiocarbamate must be increased to yield a quant. reaction with Cu. *Procedure*—Dissolve 0·1 to 1 g of a Zn, Pb or Mg alloy in conc. HNO_3 (1 to 5 ml) or 0·1 to 1 g of an Al or Sn alloy in conc. HCl (1 to 10 ml) with the addition of HNO_3 or H_2O_2, and boil off the oxides of nitrogen or the excess of oxygen. Treat the soln., or an aliquot, with 2 ml of aq. ammonium citrate (dissolve 400 g of citric acid in 500 ml of water, neutralise to litmus with 25 per cent. aq. NH_3, and dilute to 1 litre), add 10 ml of ethylenediaminetetra-acetic acid soln. (made by mixing 50 g with 50 ml of 25 per cent. aq. NH_3 and diluting with water to 250 ml), neutralise to litmus with 25 per cent. aq. NH_3 and then add 3 ml in excess. Add 10 ml of 1 per cent. aq. Na diethyldithiocarbamate and shake the soln. for 10 min. with 25 ml of $CHCl_3$. Repeat the extraction twice with 10 ml of $CHCl_3$ and dilute the combined extracts to 50 ml. Prepare a blank by the same procedure. Filter both extracts, determine the absorption of the test at 436 mμ and interpret the results from a calibration curve prepared with known quantities of Cu.

W. C. JOHNSON

1148. A rapid iodimetric method for the determination of copper and zinc. S. Z. Haider and M. H. Khundkar (*Anal. Chim. Acta*, 1955, **12** [1], 1–5).—Copper is determined iodimetrically, and the following reaction is used for the volumetric determination of Zn in the same soln.: $2K_3Fe(CN)_6 + 2KI + 3ZnSO_4 \rightarrow K_2Zn_3[Fe(CN)_6]_2 + 3K_2SO_4 + I_2$. Iodine is not liberated from KI by $K_3Fe(CN)_6$ in the absence of Zn·· if the soln. is neutral or contains only a weak acid. To avoid the reduction of $K_4Fe(CN)_6$ by the pptd. CuI, the latter is converted into CuCNS by the addition of KCNS. *Procedure*—Neutralise the soln. of Cu·· (0·02 to 0·1 g) and Zn·· (0·07 to 0·16 g) with aq. NaOH and add 1 to 2 drops of dil. H_3PO_4. Add KI ($\simeq$ wt. of Cu present), shake the vessel, add KCNS ($\simeq$ 8 times the wt. of Cu present) and titrate with 0·1 N $Na_2S_2O_3$ until most of the iodine has reacted; dilute to 50 ml, add starch soln. and complete the titration. Add 4 g of K_2SO_4 and 3 to 4 g of KI; then add 0·2 M $K_3Fe(CN)_6$ in 1-ml portions and titrate the iodine liberated by each portion with 0·1 N $Na_2S_2O_3$. Toward the end of the titration, reduce the 0·2 M

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$K_3Fe(CN)_6$ portions to 0·5 ml. The titration is complete when no blue colour is observed after a lapse of 30 sec.; 1 ml of 0·1 N $Na_2S_2O_3 \equiv 0.0099$ g of Zn (1 per cent. greater than the theoretical factor). Results are correct to within $\pm$ 0·7 per cent.

W. C. JOHNSON

1149. Determination of copper and arsenic or antimony in the presence of one another. M. R. Verma and Y. P. Singh (*J. Sci. Ind. Res., B, India,* 1954, **13** [10], 709–713).—A simple iodimetric method has been developed for the estimation of Cu·· and As··· or Cu·· and Sb··· when they are present together. It consists in complexing the metals with alkali citrate, and titration of As··· or Sb··· with standard iodine in neutral solution. After completion of the titration, the solution is acidified, the Cu complex breaks down and the iodine equivalent of Cu is liberated and be titrated. The method is rapid and is applicable for a wide range of concentrations of the constituent elements. G. C. JONES

1150. A rapid volumetric method for the determination of beryllium in beryls and associated minerals. M. Sankar Das and V. T. Athavale (*Anal. Chim. Acta,* 1955, **12** [1], 6–12).—The sample (0·5 g) of Be ore is fused with Na_2CO_3 and brought into solution by the usual method. The Be is then determined as follows. To the acid soln. add 10 ml of 20 per cent. aq. $(NH_4)_2HPO_4$, add dil. aq. NH_3 to produce a slight cloudiness, and clear the soln. with a few drops of dil. HCl. Add 10 ml of 10 per cent. aq. disodium ethylenediaminetetra-acetate (**I**) to sequester interfering elements, and sufficient ($\simeq$ 25 ml) 2 N ammonium acetate to produce a pH of 5 to 5·5 (bromocresol green). The vol. of the soln. should now be $\simeq$ 200 ml. Boil the soln. and heat it on a water bath for 30 min. to granulate the ppt. Wash the $BeNH_4PO_4.6H_2O$ by decantation, dissolve it in a small vol. of hot dil. HCl, and re-precipitate with 5 ml of **I** and 5 ml of 20 per cent. aq. $(NH_4)_2HPO_4$ in a total vol. of 100 ml. Granulate the ppt. as before, filter, wash with 2 per cent. aq. NH_4NO_3 adjusted to pH 5 by the addition of ammonium acetate; dissolve it in 15 ml of dil. $HClO_4$ (1 vol. of 60 per cent. $HClO_4$ diluted to 10 vol.) and dilute to 250 ml. Titrate 100 ml at 40° to 50° C with 0·05 M bismuthyl perchlorate (prepared by evaporating 24 g of bismuth nitrate with 6 ml of 60 per cent. $HClO_4$ until fuming and dissolving the residue in water to produce 1 litre). To indicate the end-point, add 10 ml of a saturated soln. of diallyldithiocarbamylhydrazine (prep. described) in $CHCl_3$; excess of Bi··· produces an orange colour in the $CHCl_3$ layer; 1 ml of M Bi··· $\equiv$ 25·02 mg of BeO. Results agree to within $\pm$ 0·2 per cent. with those given by standard methods. Titanium is not sequestered by **I**, but its effect can be eliminated by adding H_2O_2 before the pptn. of the phosphate. W. C. JOHNSON

1151. Chemical differentiation of beryllium and aluminium bronzes. C. Goldberg (*Chemist Analyst,* 1954, **43** [4], 95).—A 0·5-g sample is dissolved in 10 ml of dil. aq. nitric acid (1 + 1) and diluted to 250 ml. Twenty ml of 35 per cent. disodium ethylenediaminetetra-acetate solution are added, followed by conc. aq. NH_3. If the sample is a Be bronze, a white gelatinous ppt. of beryllium hydroxide will be visible. Absence of such a ppt. indicates an aluminium bronze. H. P. PAGET

1152. Qualitative detection of magnesium by some simple mono-azo dyes. I. E. Gagliardi and

M. Theis (*Z. anal. Chem.,* 1955, **144** [2], 113–115).—Several simple mono-azo dyes derived from 1-naphthol are tested as sensitive qual. reagents for Mg. Smaller amounts of Mg are detected by the colour of the solution, larger amounts by the colour adsorbed on the pptd. $Mg(OH)_2$. Other alkaline earths do not interfere. The greatest sensitivity is 0·1 μg of Mg and the highest dilution detectable is 0·67 p.p.m. (by p-nitrophenylazo-1-naphthol).

D. R. GLASSON

1153. Qualitative detection of magnesium by means of some simple mono-azo dyes. II. E. Gagliardi and M. Theis (*Z. anal. Chem.,* 1955, **144** [4], 264–272).—The colour changes of the following mono-azo dyes, when a magnesium salt is added, can be used for detection of Mg: o-, m- and p-sulphophenylazo-1-naphthols; o-, m- and p-carboxyphenylazo-1-naphthols; 2:4- and 2:6-dinitrophenylazo-1-naphthols. As little as 0·1 μg of Mg can be detected, the limit of dilution being 1 in 1,500,000. A definite mol. structure with the formation of a co-ordinate link with Mg appears to be necessary for the colour change. A. J. MEE

1154. Direct ethylenediaminetetra-acetate titration methods for magnesium and calcium. C. W. Gehrke, H. E. Affsprung and Y. C. Lee (*Anal. Chem.,* 1954, **26** [12], 1944–1948).—A limestone sample (0·5 g, 60 to 80 mesh) is dissolved in warm HCl (1 + 1) (5 ml), and the soln. is evaporated to dryness. The residue is dissolved in HCl (1 + 1) (1 ml) and the soln. is diluted with water (25 ml). After adjustment of pH to 5 to 7 with 8 per cent. NH_4Cl - 6 per cent. v/v conc. aq. NH_3 buffer, the soln. is treated with 20 per cent. aq. Na_2SO_3 (10 ml) and, after 0·5 hr., is filtered from the pptd. $CaSO_3$. The filtrate and washings (1 per cent. aq. Na_2SO_3) are made up to 100 ml; aliquots are treated with water (50 ml) and their pH values are adjusted to $\simeq$ 8 with 6·75 per cent. NH_4Cl - 57 per cent. v/v conc. aq. NH_3 buffer (5 ml). After treatment with 2 per cent. KCN, the soln. is titrated with 0·4 or 0·1 per cent. aq. disodium ethylenediaminetetra-acetate (Eriochrome black T F-241 indicator). A precision of $\pm$ 0·03 per cent. over the range 0·85 to 21·8 per cent. of MgO is given, and results compare favourably with those from a standard 8-hydroxyquinoline method. The method is extended to the determination of CaO in plant tissues. The sample (10 g) is ashed slowly (at 550° C overnight), the residue is dissolved in dil. HCl (1 + 1) (75 ml) containing HNO_3 (2 drops) and the soln. is taken to dryness. This residue is dissolved in hot dil. HCl (1 + 1) (3 ml) and water (100 ml) and, after cooling, the soln. is made up to 250 ml. PO_4''' are removed from an aliquot containing 10 to 15 mg of Ca by passing the soln. buffered to pH 3 to 4 (10 per cent. KOH), through an anion-exchange column [Amberlite IR-4B (30 g) in chloride form] at 2 to 3 ml per min. The eluate and washings (3 × 50-ml portions of water) are titrated with disodium ethylenediamine-tetra-acetate [murexide with K_2SO_4 (1 + 200) (50 mg)], after addition of 10 per cent. KOH (5 ml). Results for six animal feeding-stuffs are compared with those from a standard oxalate method; variation between the methods is -0.062 to $+0.019$ per cent., and there is a saving in time with this method. D. A. PANTONY

1155. Spectrophotometric titration of calcium and magnesium with complexone III and metal-specific indicators. P. Karsten, H. L. Kies, H. Th. J. van Engelen and P. de Hoog (*Anal. Chim. Acta,*

1955, **12** [1], 64–71).—The principles derived from theoretical bases (*Anal. Abstr.*, 1954, **1**, 1442) are applied in practice to the titration of Ca·· with disodium ethylenediaminetetra-acetate (complexone III) with murexide as indicator, and the titration of Mg·· with Eriochrome black T as indicator, both these titrations being performed in the same soln. The principles are also applied to the phthalein complexone method of Anderegg *et al.* (*Anal. Abstr.*, 1954, **1**, 1180); in this application, Ca and Mg cannot be titrated in the same solution.

W. C. JOHNSON

1156. Calcium carbide for metallurgical purposes. C. E. A. Shanahan and F. Cooke (*J. Appl. Chem.*, 1954, **4** [11], 602–611).—A preliminary report is given on work aimed at producing a slag containing calcium carbide that is liquid at iron- and steel-making temp. A method is outlined for determining the CaC_2 in mixtures and slags by measurement of the C_2H_2 evolved on the addition of 10 per cent. aq. NaCl. For CaC_2 contents of < 5 per cent., the total C is determined by combustion with Armco iron, by use of Pb_3O_4 as accelerator. The free C is determined by digestion of another sample with dil. HCl and then carrying out the combustion. The combined C is obtained from the difference between the two results.

J. M. JACOBS

1157. Colorimetric determination of zinc and copper with o-α-2-hydroxy-5-sulphophenylazo-benzylidenehydrazinobenzoic acid. R. H. Rush (*Dissert. Abstr.*, 1954, **14** [10], 1521–1522).—The use of o-α-2-hydroxy-5-sulphophenylazobenzylidene-hydrazinobenzoic acid as a new colorimetric reagent for Zn and for Cu is described. The sensitivity for Zn is 1 pt. in 50,000,000. An absorption maximum occurs at 620 mμ for the Zn complex and at 600 mμ for the Cu complex; the reagent itself has maximum absorption at 480 mμ and very little at 600 to 620 mμ. The reagent and the Cu complex are stable at pH 5 to 10; the Zn complex is stable at pH 8·5 to 9·5. Both complexes have a reagent to metal ratio of 1 to 1 and conform to Beer's law over the range 0·1 to 2·4 p.p.m. The reactivity of this reagent towards 79 ions is reported. The separation of Zn and of Zn and Cu from interfering ions with an anion-exchange resin is discussed and results are given; these show a standard deviation of about 3 per cent. The possible application of this reagent to the determination of Co and Ni is indicated.

S.C.I. ABSTR.

1158. Separation of zinc from cadmium with special reference to the determination of zinc in cadmium metal. E. R. Baggott and R. G. W. Willcocks (*Analyst*, 1955, **80**, 53–64).—The behaviour of Cd and Zn in iodide soln. towards an anion-exchange resin is investigated and it is shown that these metals can be separated by passing a mixture of their sulphates in mixed sulphate - iodide soln. through a column of De-Acidite FF ion-exchange resin in the sulphate form. The column is first flooded with a KI - H_2SO_4 soln. and then the sample soln. (prepared by dissolving the metal in HNO_3, fuming with H_2SO_4 and diluting) is run through. Finally the column is washed with a KI soln. Zn is determined in an aliquot part of the eluate by extraction of the Zn - dithizone complex in CCl_4 and determining its optical density; the concn. of Zn is then ascertained from a previously prepared calibration graph. The eluate contains a small amount of Cd but insufficient to affect the final determination of Zn. Under the conditions

of the method Cu, Fe and Pb do not interfere. Zn can be determined in cadmium metal at a concn. of 0·005 per cent.

A. O. JONES

1159. Derivative polarography. II. Derivative polarography of cadmium, antimony and lead ions. T. Isshiki, Y. Mashiko and S. Tsukagoshi (*Pharm. Bull., Japan*, 1954, **2** [3], 263–265).—The method described earlier (*Anal. Abstr.*, 1955, **2**, 1106) is applied to Cd··, Sb··· and Pb·· ions in 0·2 N HCl soln. The results show that the max. current in each derivative curve occurs at the half-wave potential of the particular ion. The relation between concn. and max. current is not strictly linear.

H. F. W. KIRKPATRICK

1160. An indirect colorimetric method for the determination of barium. II. Micro-analysis of barium. T. Nozaki (*J. Chem. Soc. Japan*, 1954, **75** [2], 168–170).—A small amount of barium (1 to 0·01 mg) is pptd. by ammonium molybdate in ammonium acetate soln., at pH 7·6, on a micro scale. The ppt. is filtered, dissolved in HCl (1 + 2) and made up to a definite vol. A portion of the HCl soln. is treated with aq. KCNS, the red thiocyanate is extracted by ether and the amount of Mo is determined colorimetrically. Ca or Sr does not affect the result, provided the amount is not more than that of barium.

K. SAITO

1161. Quantitative inorganic chromatography. VI. Chromatography of barium and strontium. A. Murate (*J. Chem. Soc. Japan*, 1954, **75** [2], 135–139).—Filter-paper is impregnated with alumina by treating it with a soln. of sodium aluminate (0·3 M) and sodium bicarbonate, successively, at 50° C. Bands of Ba and Sr are detected by Na rhodizonate; the width of the adsorption band ($\simeq 10^{-8}$ mole, in Cl' or NO_3' soln.) is proportional to the concn., which enables rough quant. analysis of both elements. The width of adsorption bands of ions (4×10^{-3} M) increases in the following order: H < Cu < Cd < Ba $\simeq$ Zn < Sr < Co < Ni < Mn (in NO_3' soln.); H < Cu < Zn < Cd < Ni = Co < Mn (in SO_4'' soln.). This order can be related to the increasing solubility of their hydroxides, showing that the ease with which they are adsorbed decreases in the same order. The formation of bands appears to be due to the deposition of metal hydroxide, and also carbonate for Ba and Sr. In the combinations Ba - Cu, Sr - Cu, Zn - Cd, Sr - Zn and Sr - Ba, in spite of the satisfactory separation of bands, only the more preferentially adsorbed constituent (*e.g.*, Cu, Cu, Cd, Zn and Ba, respectively) can be determined.

K. SAITO

1162. Isotopic analysis of some gaseous boron hydrides by thermal conductivity. W. S. Koski, P. C. Maybury and J. J. Kaufman (*Anal. Chem.*, 1954, **26** [12], 1992–1994).—Deuterated boron hydrides are decomposed at 500° to 800° C, and the resulting H to D ratio is determined by thermal-conductivity measurements. Alternatively, for pure B_2H_6 and B_5H_9, the ratio is measured directly on those gases by comparison of their conductivities with those of the normal isotopic samples. The balancing voltage ratios are interpreted by means of a calibration curve.

D. A. PANTONY

1163. Direct standard analytical determination of aluminium by ethylenediaminetetra-acetic acid (complexone III). M. Theis (*Z. anal. Chem.*, 1955, **144** [2], 106–108).—Al is directly titratable with

complex[...] in hot [...] determin[...] a small [...] solution [...] good ac[...] Ca··, M[...]

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1165. [...] A. Calc[...] —Whe[...] dipicry[...] develop[...] dioxan [...] the pre[...] in a gla[...] a knob[...] knob is [...] If, on p[...] the dro[...] may be[...] solution [...] of the [...] should [...] result i[...] alumin[...] amine.[...]

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complexone III and use of Chromazurol S indicator in hot solution at pH 4. The end-point can be determined either directly or on back-titration of a small excess of the complexone with the aluminium solution after addition of sodium acetate. Very good accuracy is attained in the presence of $Fe^{...}$, $Ca^{..}$, $Mg^{..}$, $Na^{.}$, $K^{.}$, $NH_4^{.}$, $Mn^{..}$ and $TiO^{..}$.

D. R. GLASSON

1164. Determination of small amounts of aluminium in steel by a spectrochemical method. B. C. Kar (*J. Sci. Ind. Res., B, India*, 1954, **13** [11], 855–856).—An investigation into a spectrochemical method for determining small amounts of Al in steels by means of a medium quartz spectrograph and synthetic standards is described. Standard solutions containing 0·005 to 0·20 per cent. of Al were prepared by dissolving separately spectroscopically pure Fe, Mn and Al in pure hydrochloric acid and then mixing. Nitric acid was added to oxidise Fe^{II} to Fe^{III}. Drillings of test samples were similarly ·dissolved and oxidised. Spectra of the standard and test solutions were taken by dropping one to two drops of the test solution on the pre-sparked flat top of the lower graphite electrode of the spectrograph and passing the spark when the drops were dry. Photometric measurements were made with a non-recording photo-electric microphotometer. Working curves were drawn by plotting

log (microphotometer deflection of Al line)

log (microphotometer deflection of Fe line)

against the known percentage of Al in the standard solutions. The percentage of Al in the unknown solution was determined by interpolation.

G. C. JONES

1165. Spot test for hydrous solids and alumina. A. Caldas (*Chemist·Analyst*, 1954, **43** [4], 100–101). —When H_2O is added to a 1 per cent. solution of dipicrylamine in anhyd. dioxan, an orange colour develops, probably owing to the formation of the dioxan salt of the dipicrylamine anion. To detect the presence of H_2O in a solid, the sample is inserted in a glass tube which is closed by a stopper having a knob protruding into the tube. On the end of the knob is suspended a drop of the reagent solution. If, on gentle heating of the tube, water is evolved, the drop turns orange. Alternatively, the sample may be put on a spot plate and covered with the solution; the orange colour develops at the surface of the sample if it contains H_2O. A blank test should be observed simultaneously. A positive result in the second method is given by anhydrous alumina owing to chemisorption of the dipicrylamine.

H. P. PAGET

1166. Characterisation of hydrated aluminas by infra-red spectroscopy. Application to study of bauxite ores. L. D. Frederickson, jun. (*Anal. Chem.*, 1954, **26** [12], 1883–1885).—The hydrated alumina forms, boehmite, gibbsite, diaspore and bayerite, are examined by infra-red spectrophotometry, while supported in KBr or liquid paraffin. Variation in O–H bond stretching and deformation characteristics enable the forms to be differentiated. Energy - wavelength diagrams are presented for the pure forms and for several bauxite and diaspore ores in which the various species are identified, unless present in small amounts. The work is supported by chemical analysis and, particularly, X-ray diffraction and spectrographic analysis.

D. A. PANTONY

1167. Detection of gallium by means of o-salicylideneaminophenol. V. Patrovský (*Chem. Listy*, 1954, **48** [4], 537–538).—A study of the reactions of o-salicylideneaminophenol (**I**) with In, Ga, Ge and Sc has shown that only Ga reacts, giving a yellow fluorescence with a green tint in weakly acidic solutions (pH 4·5 to 5·5). A similar fluorescence, given with **I** by Al, can be screened with $NaBF_4$. *Procedure*—An HCl soln. of the sample containing Ga, buffered with Na acetate and acetic acid, is treated with a 0·05 per cent. soln. of **I** in ethanol (0·5 ml) and with a few ml of aq. $NaBF_4$ (prepared by dissolving 1·8 g of cryst. borax and 3·2 g of NaF in 80 ml of H_2O acidified with acetic acid and diluting to 100 ml). The fluorescence is clearly observed in dilutions of $\not>$ 0·08 mg of Ga per 50 ml. Procedures are given for the detection of Ga in aluminium, bauxite, silicates and sphalerite.

G. GLASER

1168. The determination of gallium. G. W. C. Milner (*Analyst*, 1955, **80**, 77–78).—Ga forms a stable complex with ethylenediaminetetra-acetic acid (**I**) that serves for its volumetric determination, and the lake formed with gallocyanine can be used to show the end-point. The Ga soln. is treated with 10 ml of glacial acetic acid, and conc. aq. NH_3 is added in drops to adjust the pH to 2·8 as shown by a direct-reading pH meter. The indicator, 5 drops of a saturated soln. of gallocyanine in glacial acetic acid, is added and the mixture is stirred to promote formation of the blue lake. The liquid is then titrated with 0·02 M or 0·002 M **I** (according as the Ga present is more or less than 2 mg) with stirring until the colour of the lake is discharged (1 ml of 0·02 M soln. $\equiv$ 1·394 mg of Ga). The method is suitable for the determination of 0·25 mg to 50 mg of Ga and is applicable after extraction of Ga with ether as previously reported (Milner *et al.*, *Anal. Abstr.*, 1954, **1**, 2064). The standard solutions of **I** are prepared by dilution of a 0·1 M soln. prepared and standardised as previously reported (Milner *et al.*, *Anal. Abstr.*, 1954, **1**, 2360). Recovery of Ga is good.

A. O. JONES

1169. Quantitative analysis of indium. D. Jentzsch, I. Frotscher, G. Schwerdtfeger and G. Sarfert (*Z. anal. Chem.*, 1955, **144** [1], 8–16).—A procedure for the quant. determination of In in the presence of Pb, Ag, Sn, Bi, Fe, Ge, Ga, Al, Mn, Cd, Cu, Zn, Ni, Co and As is described. Preliminary treatment with aqua regia, H_2SO_4 and aq. NH_3 separates all except Sn, Fe, Al, Mn, Cu and As. The ion-exchange resin Wofatit L 150, in the presence of 5 N HCl, separates In and Sn from the remaining elements; the separation is controlled spectrographically. After elution of In and Sn with 0·1 N HCl and treatment with aq. NH_3 and tartaric acid, the In is determined polarographically with a relative accuracy of $\pm$ 8 per cent. for indium contents of 0·005 to 0·03 per cent. and about $\pm$ 3 per cent. for In content of about 0·1 per cent.

D. R. GLASSON

1170. Spectrophotometric determination of combined carbon in iron. H. Newberg (*Chemist Analyst*, 1954, **43** [4], 93).—A 0·5-g sample is treated with 20 ml of dil. nitric acid (1 + 2). After gentle heating, 4 drops of hydrofluoric acid are added and the solution is then heated strongly until solution is complete; it is then cooled and diluted to 250 ml. After filtration, the optical density is measured at 375 mμ, by using dil. nitric acid (1 + 20) as reference solution. A calibration curve is prepared

from samples of known carbon content. The texture of the filter-paper is unimportant. Mo causes interference with the colour.　　H. P. PAGET

1171. Storage of sea-water samples for the determination of silicate. J. B. Mullin and J. P. Riley (*Analyst*, 1955, **80**, 73–74).—In the study of variations in the silica content of ocean waters, stored samples must be protected from contamination by extraneous SiO_2. The effect of pH on the rate of uptake of Si by sea-water from soda glass, Pyrex glass and transparent silica bottles has been investigated. Both acidified and unacidified waters in all bottles showed fairly regular weekly increases in Si content. The attack of unacidified sea-water on soda glass was $\simeq 13$ times as rapid as on silica glass and $\simeq 4$ times as rapid as on Pyrex glass, but there was variation from bottle to bottle either can be used for storage of acidified samples (pH 2·5) for short periods. In polythene bottles, filtered and sterilised sea-water enriched with SiO_2 (> 15 mg of Si per litre) showed very little change in silica content (< 2 per cent.) in three months. Similarly treated samples with the normal content of silicate (0·5 mg of Si per litre) generally decreased considerably (up to 20 per cent.) for one week of storage and then remained constant. Filtered acidified sea-water samples (pH 2·1 to 2·5), stored in polythene bottles in the dark, remained constant to within 1 per cent. for at least 5 weeks (15 mg of Si per litre). Suspended silicious matter should be removed from sea-water before storage. A simple device for filtering sea-water during collection at sea is described.　　A. O. JONES

1172. Separation of titanium combined with spectrophotometric determination of titanium in steel. J. R. Simmler, K. H. Roberts and S. M. Tuthill (*Anal. Chem.*, 1954, **26** [12], 1902–1904).— Steel turnings (1 g) containing 0·05 to 1 per cent. of Ti are digested with dil. (1 + 1) HCl (20 ml) until a crust forms, and the residue is taken up in hot dil. (1 + 3) HCl (40 ml). After filtration, the soln. and washings (1 per cent. HCl), together with the soln. obtained by igniting the residue and treating it with sulphuric acid plus hydrogen fluoride, evaporating and fusing with $KHSO_4$ and leaching it with dil. (1 + 9) sulphuric acid, are made up to 100 ml. A 10-ml aliquot and a reagent blank (10 ml of N HCl) are treated separately as follows: the suspensions obtained after addition of 1 per cent. Zr^{IV} soln. [2·78 per cent. $Zr(SO_4)_2.4H_2O$ in dil. (1 + 4) HCl] (1 ml) and 20 per cent. As^V soln. [20 per cent. aq. As_2O_5] (1 ml) are centrifuged; the supernatant liquors are treated with further 1 per cent. Zr^{IV} soln. (1 ml), when, after centrifuging, the liquid is rejected. The ppt. are washed with 0·2 per cent. As^V soln. in HCl (1 + 9) and are dissolved in 96 per cent. H_2SO_4 (2·5 ml); the soln. plus the H_2SO_4 (1 + 9) washings are made up to 25 ml with water. The soln. is treated with 30 per cent. H_2O_2 (1 drop) and the colour is measured at 410 $m\mu$ vs. H_2SO_4 (1 + 9). After allowance for the reagent blank, Ti concn. is obtained from a calibration curve derived from standard Ti^{IV} (from TiO_2) soln. The zirconium arsenate pptn. allows the complete separation of Ti from Mo, V and Cr. Analytical figures for six standard steels are given.　　D. A. PANTONY

1173. Benzoin as a fluorescent reagent for the detection of germanium. N. Appala Raju and G. Gopala Rao (*Nature*, 1955, **175**, 167).—A fluorescent test for the detection of germanium by the use of a benzoin reagent is described. *Procedure*—To 5 ml of reagent, prepared by shaking an excess of benzoin $[C_6H_5COCH(OH)C_6H_5]$ with 90 per cent. ethanol at 28° C and filtering, is added 1·0 ml of a slightly alkaline solution of Ge as germanate. After the mixture has been made up to 10 ml with 90 per cent. ethanol it is exposed in a quartz test-tube to the filtered ultraviolet light from a "Cenco" black-light source operated on 220-volt a.c. mains. Even with 10 μg of germanium, a greenish-yellow fluorescence is observed. Sensitivity is 1 in 100,000. Comparison is made with the resacetophenone reagent.
　　C. H. WHITTON

1174. An indirect colorimetric method for the determination of zirconium. T. Nozaki (*J. Chem. Soc. Japan*, 1954, **75** [5], 582–586).—An indirect colorimetric determination of Zr is proposed which depends on the low solubility of zirconium phosphate in acidic soln. The Zr (1·7 to 0·002 mg) is pptd. as phosphate from 8 per cent. aq. H_2SO_4 (when Ti is present, H_2O_2 is added), and washed thoroughly with aq. NH_4NO_3 soln. on a micro scale. The ppt. is dissolved by adding N KOH and saturated Na K tartrate soln. It is neutralised to p-nitrophenol and the phosphate-ion concn. is determined colorimetrically by the usual method. $Al^{...}$ ($\geqslant 720$ times that of Zr), $Fe^{...}$ ($\geqslant 70$ times), $Ti^{...}$ ($\geqslant 48$ times) and $Th^{...}$ ($\geqslant 88$ times) do not affect the result. When more Ti is present, Zr can be re-pptd. from the tartrate soln. When the amount of $Fe^{...}$ is > 70 times that of Zr, it must be removed by ether extraction before the pptn. of Zr. The error appears to be mainly due to the different contents of Hf in the Zr sample.
　　K. SAITO

1175. Polarographic determination of tin in ores. L. Bouberlova-Kosinova (*Věst. Ústřed. Ústavu Geol.*, 1954, **29** [1], 13–20; *Referativnyǐ Zh., Khim.*, 1954, Abstr. No. 27,551).—The tin is oxidised to Sn^{IV} and determined polarographically in HCl - NaCl soln. (optimum concn. of Cl′, 5 to 10 N), the second reduction wave ($Sn^{..}/Sn$) of $E_{\frac{1}{2}} = -0·62$ V being used. Silica (up to 80 per cent.), Al, Mn, Ca, Mg, Na, Li, Zn, Cr, Ni, Fe, Mo, Bi, Cu and Sb do not affect the determination, but Tl, W and Pb, and to a lesser extent Ti and As, interfere; V and Ge distort the wave. Tin is separated from interfering ore components by pptn. as a complex with disodium ethylenediaminetetra-acetate in ammoniacal soln. For tin - tungsten ores, a ground sample is evaporated to dryness with dil. HNO_3; the residue is dissolved in aqua regia on a water bath and the soln. is evaporated to dryness; HCl is added and, after evaporation to dryness, the residue is dissolved in HNO_3. The soln. is neutralised with NH_3 until a faint ppt. appears, which is redissolved with a few drops of HNO_3. Excess of a solution of disodium ethylenediaminetetra-acetate is added, and then aq. NH_3 soln. until the soln. is strongly alkaline. The pptd. complex is collected on a fine-grain filter and fused in an iron crucible with 20 g of Na_2O_2; the filter is ignited separately and the residue is added to the melt. The product is dissolved in water and the solution is made up to 250 ml; 25 ml of this soln. are mixed with 1 ml of 0·5 per cent. gelatin soln. and the mixture is made up to 100 ml with conc. HCl. The soln. is then polarographed.

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For the determination of tin in other ores, the sample is fused with Na_2O_2 and the analysis is carried out as described. E. HAYES

1176. The micro-volumetric determination of lead with thionalide and its application to the analysis of tin-base and lead-base alloys. C. Cimerman and M. Ariel (*Anal. Chim. Acta*, 1955, **12** [1], 13–28).— Lead is determined by pptn. with thionalide followed by iodimetric titration of the thionalide in the ppt. *Procedure*—To the slightly acid soln. (1 to 5 ml) of Pb (1 to 10 mg) as nitrate, add 50 mg of Na K tartrate and 1 drop of 1 per cent. ethanolic phenolphthalein, and neutralise with 1 to 2 drops of conc. aq. NH_3. Add 100 mg of KCN and dilute the soln. with an equal vol. of 2 N Na_2CO_3; then add dropwise, with stirring, a freshly prepared soln. of 40 to 50 mg of thionalide in 1 to 2 ml of acetone. Heat the soln. cautiously to boiling and filter immediately with low suction through a filter-stick of medium porosity. Wash the ppt. with two 2-ml portions and three 1-ml portions of acetone - water mixture (1 + 1) and then with two 2-ml portions of water. Dissolve the ppt. in 4 to 6 ml of a mixture of acetic acid and 2 N H_2SO_4 (3 + 1) and wash the apparatus with one 2-ml and three 1-ml portions of the same mixture. Bubble CO_2 through the soln., add 20 mg of KI and, if $\not>$ 5 mg of Pb are present, titrate with 0·01 N KIO_3 - KI (prepared by dissolving 0·3567 g of KIO_3 in 200 ml of water containing 1 g of NaOH and 2 to 3 g of KI and diluting to 1 litre) until the free iodine is apparent. If > 5 mg of Pb are present, use 0·02 N KIO_3 - KI prepared similarly, but with 0·7134 g of KIO_3. Dilute the soln. with twice its vol. of water, add 1 ml of 1 per cent. starch soln. and titrate the excess of I with 0·01 N $Na_2S_2O_3$. Deduct 0·01 ml from the vol. of 0·01 N KIO_3 - KI consumed (1 ml of 0·01 N KIO_3 - KI $\equiv$ 1·036 mg of Pb). The method is adapted to the determination of Pb in Sn-based and Pb-based metals and to alloys containing 5 to 72 per cent. of Pb together with Sn, Sb and Cu. In such applications, a preliminary separation of the Pb as $PbSO_4$ is necessary. The $PbSO_4$ is dissolved in aq. ammonium acetate and the general procedure is followed, except that the Pb - thionalide compound is pptd. from a hot soln. The max. error is $\pm$ 0·5 per cent. of the amount of Pb present. W. C. JOHNSON

1177. A contribution to lead determination. C. Mahr and H. Otterbein (*Z. anal. Chem.*, 1955, **144** [1], 28–30).—Lead is selectively pptd. by thiourea in HNO_3 (+ HCl) solution at $0°$ C, so permitting determination of Pb in the presence of Sn and Sb. After decomposition of the ppt. by hot water to $Pb(NO_3)_2$ and thiourea, the Pb is determined by indirect titration with complexone III, the excess of the latter being titrated with a magnesium salt solution; Eriochrome black T indicator then gives a sharp end-point. Addition of KCN masks foreign metals, and tartrates buffer the solution. Amounts of 5 to 77 mg of Pb are determined to a mean accuracy of $\pm$ 0·13 per cent. within extremes of $\pm$ 0·6 per cent. and amounts of 1 to 5 mg to within $\pm$ 2 per cent. D. R. GLASSON

1178. Thorium: its estimation and separation from cerite earths. Use of aryloxyacetic acids. N. Eswaranarayana and Bh. S. V. Raghava Rao (*J. Sci. Ind. Res., B, India*, 1954, **13** [9], 657–659). —The use of *o*-chlorophenoxyacetic acid, *p*-chlorophenoxyacetic acid and *p*-chloro-*m*-tolyloxyacetic acid as precipitating agents for thoria in the separation of Th from cerite earths has been investigated.

With each reagent, separation in a single precipitation is possible when the proportion of cerite earths in the solution is 2 to 4 times that of Th. The optimum pH for precipitation is 2·5 to 3·6. As little as 3·5 mg of thoria can be estimated and *p*-chlorophenoxyacetic acid is the most effective of the reagents tested. G. C. JONES

1179. Direct volumetric procedure for estimation of thorium and its application in the preparation of thorium reagent for micro-estimation of fluorine. P. Venkateswarlu, A. N. Ramanathan and D. Narayana Rao (*Indian J. Med. Res.*, 1953, **41** [2], 253–255).—To 20 ml of 0·1 N oxalic acid is added 1 ml of 0·5 per cent. aq. alizarin red S, followed by $\simeq$ 20 ml of water, and the $\simeq$ 0·1 N solution of $Th(NO_3)_4$ is run in until the yellow colour turns to pink. For 0·01 N solutions, the end-point is sharper if the final stage of the titration is carried out at 80° C. The results obtained by this method were found to be in good agreement with those of conventional gravimetric determinations. This volumetric method for the estimation of Th provides a convenient means for the preparation of Smith and Gardner's reagent for the micro-estimation of F (*cf.* Atomic Energy Commission Unclassified Document UR-36). An $\simeq$ 0·1 N $Th(NO_3)_4$ soln. is standardised by the above method and an aliquot containing 0·134 g of $Th(NO_3)_4$. $4H_2O$ is treated with 7·5 ml of N HCl and made up to 1 litre with fluorine-free water (1 ml $\equiv$ 5 μg of F). J. M. JACOBS

1180. Determination of thorium with organic reagents. Uses of some aryl fatty acids. S. K. Datta and G. Banerjee (*Anal. Chim. Acta*, 1955, **12** [1], 38–46).—Thorium is pptd. quant. from boiling soln. buffered with ammonium acetate and adjusted to pH 2·8 to 5·2, by addition of hot 1 per cent. aq. phenylacetic acid. The voluminous ppt. coagulates and is filtered off while hot, washed with a 0·1 per cent. soln. of the reagent containing 0·5 per cent. of ammonium acetate, then washed with hot water and finally ignited to ThO_2. At pH 3·2 to 5·2, ThO_2 (4·4 mg) can be separated quant. from a 16-fold excess of CeO_2 plus La_2O_3 by one pptn., or from a 30-fold excess by double pptn. Satisfactory separations from $Ca^{..}$, $Ba^{..}$, $Sr^{..}$, $Zn^{..}$, $Al^{...}$, $Cr^{...}$, $Ti^{....}$, $Ni^{..}$, $Co^{..}$ and $Mn^{..}$ are also effected in solutions of pH 4·4 to 4·8, but the last three require a double pptn. Uranium in large excess is also separated by a double pptn. Iron and zirconium interfere. The composition of the ppt. is $(C_6H_5CH_2COO)_3Th(OH)$, but it is not sufficiently constant for direct gravimetric purposes. W. C. JOHNSON

1181. Colorimetric micro-determination of nitrogen dioxide in the atmosphere. B. E. Saltzman (*Anal. Chem.*, 1954, **26** [12], 1949–1955).—Gas samples containing NO_2 (4000 ml for < 1 p.p.m., 30 to 250 ml for 1 to 100 p.p.m.) are collected by standard procedures, the gases being absorbed in 0·5 per cent. sulphanilic acid and 0·002 per cent. N-(1-naphthyl)ethylenediamine dihydrochloride in 14 per cent. acetic acid. Colour development is completed within 15 min. and is compared with standards prepared from $NaNO_2$, either by eye or spectrophotometrically at 550 mμ. Ozone interferes and should be removed by passing the gas over prepared MnO_2; SO_2 destroys the colour when its concn. is > 10 times that of NO_2 and its effect can be minimised by the addition of acetone or CrO_3 to the absorbing system. Other nitrogen oxides, H_2S and formaldehyde do not interfere, but Cl and H_2O_2 affect the colour. D. A. PANTONY

1182. Analysis for industry. [Determination of nitrate.] M. Williams (*Ind. Chem.*, 1954, **30**, 594–595).—Analytical methods for the gravimetric determination of nitrate are described. Special reference is made to the use of nitron, di-(1-naphthylmethyl)amine acetate, cinchonamine, diethylamino-1-phenylethyl-*p*-[nitro]benzoate, 6-ethoxy-5-nitroquinoline, *p*-tolyl*iso*thiourea, N-benzyhydryldiethylamine and di*cyclo*hexylthallium.　　D. R. PECK

1183. Method of investigation of nitrates in chroming baths. J. Ibarz Aznárez and J. B. Vericat Raga (*An. Soc. Esp. Fis. Quím., B*, 1954, **50** [12], 985–988).—The determination of HNO_3 by reaction with $Fe(NH_4)_2(SO_4)_2$ and with brucine in $CHCl_3$ is examined. The sensitivity of the first method is 0·5 g per litre, reducing to 0·132 per cent. when the large quantities of CrO_3 present in chroming baths have to be removed by pptn. as $BaCrO_4$. The sensitivity of the second method (Haase, *Chem. Z.*, 1926, **50**, 372) is 0·4 mg per litre for HNO_3 solutions, reducing to 20 mg per litre when CrO_3 has to be pptd. The decrease must be due to retention of some of the HNO_3 on the $BaCrO_4$ ppt.; however, in the absence of HNO_3, a positive reaction is given by traces of chromate, the colour having a light violet tint.　　D. LEIGHTON

1184. Spectrographic analysis. III. Quantometric determination of phosphorus in low alloy iron. J. M. Gillette (*Dissert. Abstr.*, 1954, **14** [10], 1518–1519).—In the spectrographic determination of P in Fe alloys, the 1·5-metre grating in the quantometer does not sufficiently resolve the phosphorus line from the copper line close by. A special filtering device has been designed to overcome this difficulty. It consists of a small 60° quartz prism placed just behind the secondary slit of the quantometer to separate the visible and u.v. rays, a narrow secondary aperture being used to block the deviated visible radiation. An arc-like multi-source discharge is used with a 60-sec. integration period to bring out the phosphorus line. The precision of the phosphorus determination is $0·083 \pm 0·004$ per cent. of P; the limit of detection at the 25 per cent. error level is 0·55 per cent. of P. There is some interference from Cu, for which a correction can be made.　　S.C.I. ABSTR.

1185. Electrical mobility of phosphate ions in paper electrochromatography. J. L. Engelke and H. H. Strain (*Anal. Chem.*, 1954, **26** [12], 1872–1874).—The ions H_2PO_4', HPO_4'' and PO_4''', mixed with radio-tracers, are examined electro-osmotically at various pH values in 0·1 M lactic acid soln.; 0·1 $M \cdot H_2O_2$ or hydrazine is used as indicator. In general, mobilities of H_2PO_4' and HPO_4'' are roughly equal and are about one-quarter that of PO_4'''; in strongly acid soln., the migration of phosphate is towards the anode and in soln. of higher pH value towards the cathode. The mechanism of the electro-osmotic flow is discussed.　　D. A. PANTONY

1186. Volumetric determination of peroxydiphosphate. T. Chulski (*Dissert. Abstr.*, 1954, **14** [11], 1904).—The preparation of lithium peroxydiphosphate (**I**) used in this determination is described in detail. Peroxydiphosphate [peroxypyrophosphate] (**II**) is determined by adding excess of Fe·· and back-titrating the excess with $K_2Cr_2O_7$ soln., sodium diphenylaminesulphonate being used as indicator. Reaction mixtures 0·25 to 1·0 M in H_2SO_4 and 0·25 to 2·0 M in $HClO_4$ give satisfactory results; results were less satisfactory in 1·0 to 2·0

M HCl. The amount of Fe·· is not critical, and phosphate can be present. Potentiometric titration with ferrous ammonium sulphate (**III**) gives satisfactory results in 0·25 to 0·50 M H_2SO_4 or 0·50 M H_3PO_4. The equivalence point in the titration of **II** against **III** is successfully determined by means of the dead-stop end-point technique. Results are good if the soln. of **II** is 0·25 to 1·0 M in H_2SO_4, 0·50 to 1·0 M in H_3PO_4 or 0·25 to 0·50 M in $HClO_4$. An iodimetric titration gives satisfactory results both in the absence and presence of phosphate; the solution of **II** is 1·0 M in H_2SO_4. The rate of reaction in this determination is influenced by the pH of the mixture; a small increase in pH greatly increases the rate of reaction.　　S.C.I. ABSTR.

1187. A new qualitative test for vanadium. R. Přibil and J. Michal (*Chem. Listy*, 1954, **48** [4], 621–622).—When a soln. containing V at pH 6 to 7 is shaken with a 0·5 per cent. soln. of quercetin in a (1 + 1) mixture of ether and acetylacetone, the organic layer turns an intense red colour. The reaction is extremely sensitive, 2 μg of V in 1 ml being detectable. The disturbing effect of Fe can be masked by the addition of complexone IV (disodium 1:2-diamino*cyclo*hexanetetra-acetate). A soln. of quercetin in acetone turns green in the presence of V and is also suitable for the detection of the metal.　　G. GLASER

1188. The determination of vanadium by means of selenous acid. B. Suseela (*Z. anal. Chem.*, 1955, **144** [5], 329–331).—Vanadyl selenite ($VOSeO_3.3H_2O$) is quant. precipitated by selenous acid from a solution of vanadyl sulphate containing ammonium acetate in 50 per cent. ethanol. The precipitate is dissolved in conc. HCl solution and the liberated selenous acid is titrated iodimetrically. To a solution of vanadyl sulphate, add an excess of selenous acid solution (about 0·1 N) followed by ethanol to a concentration of about 50 per cent. Add 5 per cent. sodium acetate solution until the pH lies between 4 and 4·5. Filter off and wash the precipitate, dissolve it in conc. HCl solution and make up to 250 ml. To a suitable aliquot, add excess of KI solution and titrate the liberated iodine with 0·1 N sodium thiosulphate. One ml of 0·1 N $Na_2S_2O_3 \equiv 0·001274$ g of V.　　P. S. STROSS

1189. Photometric determination of vanadium by means of catechol. V. Patrovský (*Chem. Listy*, 1954, **48** [4], 622–624).—A new method for the colorimetric determination of V is based on the formation of an intense blue coloration of V^{IV} with catechol, which is clearly discernible in concn. as low as 0·08 mg of V in 100 ml. The following metals interfere: Cu, Ni, Co and Cr or Fe and Mn if present in quantity. Aluminium, if present in large amounts, separates in the neutral soln. as the hydroxide, and is best removed as the chloride by a current of HCl gas (Swift, *J. Amer. Chem. Soc.*, 1924, **46**, 2375). *Procedure.*—Treat the soln. containing V at pH 4 to 8 with 10 per cent. aq. catechol (10 ml), 10 per cent. aq. $Na_2SO_3.7H_2O$ (10 ml) and $\simeq 17$ per cent. aq. NH_3 (10 ml), dilute to 50 ml and, after 5 min., measure the extinction through a yellow filter; max. absorption takes place at approx. 580 mμ. The method is especially suitable for the determination of traces of V in ores and minerals.　　G. GLASER

1190. The precipitation of bismuth with arsanilic acid. A. Musil and R. Pietsch (*Z. anal. Chem.*, 1955, **144** [5], 347–350).—Bismuth is quant. precipitated from HNO_3 soln. at pH 2 to 3 by the use

of arsanilic acid, only a small excess of which is necessary. The precipitate is dried at 140° to 170°C. Precipitation is also possible in H_2SO_4 soln., but for quant. results the precipitate must be re-dissolved and re-precipitated from HNO_3 soln. Sodium, potassium, ammonium and acetate ions do not interfere, but chloride, phosphate and tartrate must be absent.   P. S. STROSS

1191. Titration of bismuth with ethylenediamine-tetra-acetic acid. J. S. Fritz (*Anal. Chem.*, 1954, **26** [12], 1978–1980).—A soln. (10 to 15 ml) containing 30 to 80 mg of Bi··· is warmed in the presence of thiourea (0·5 to 0·8 g). The soln. is treated with almost enough aq. NH_3 to cause pptn. and the pH is adjusted to 1·5 to 2 with dil. $HClO_4$ when the vol. is 40 to 50 ml. The mixture is titrated with 0·05 M disodium ethylenediaminetetra-acetate [standard-ised against Zn (Eriochrome black T)] to the dis-appearance of the yellow colour. Fe··· and Sb··· can be rendered innocuous with ascorbic acid (0·3 to 0·4 g) and tartrate, respectively. Thirty-six ions were tested and only oxalate, PO_4''', Ni··, Sn····, VO·· and Zr···· interfered.   D. A. PANTONY

1192. Compleximetric titration of bismuth. Kuang Lu Cheng (*Anal. Chem.*, 1954, **26** [12], 1977–1978).—A soln. ($\simeq$ 50 ml) containing 10 to 50 mg of Bi··· is adjusted to pH 1·5 to 2 (dil. aq. NH_3 or HNO_3) and a known (excess) vol. of 0·01 M disodium ethylenediaminetetra-acetate standardised against a 0·01 M zinc solution is added. The excess is back-titrated with standard 0·01 M Bi··· [0·5 per cent. KI (10 ml) indicator], allowance being made for the indicator blank. In the presence of Cu·· or Ni··, the Bi··· is titrated directly with ethylene-diaminetetra-acetate under the same conditions. The effect of thirty-eight extraneous ions is listed and of these, Tl·, Ce···, Fe···, Ti···, Zr····, Hg··, Ag·, Mo······ and VO_3' interfere. D. A. PANTONY

1193. Polarographic determination of small amounts of bismuth in copper. Z. Zábranský (*Chem. Listy*, 1948, **48** [4], 617–618).—The method consists in the polarographic determination of Bi, after separating it from Cu by extraction with dithizone (I). *Procedure*—Dissolve 5 g of copper shavings, containing at least 0·0005 per cent. of Bi, in conc. HNO_3 (50 ml) and evaporate the soln. with conc. H_2SO_4 (8 ml) until white fumes of SO_3 are evolved. Cool, add H_2O (50 ml) and 50 per cent. citric acid (1 ml), followed by conc. aq. NH_3 until the ppt. that forms redissolves. Mix the soln. with 50 per cent. KCN (50 ml), dilute to 200 ml, extract with a 0·5 per cent. soln. of I in chloroform (6 × 3 ml), evaporate the combined extracts to dryness, heat the residue with conc. H_2SO_4 (0·5 ml) and 30 per cent. H_2O_2 (to destroy organic matter), expel the excess of H_2O_2, cool, dilute with 2 M Na acetate (5 ml) containing 0·05 M complexone III and polarograph. Under the given conditions, the $E_{\frac{1}{2}}$ value of Bi is approx. − 0·6 V and that of Pb (also extracted by I) − 1·1 V.   G. GLASER

1194. Determination of bismuth in metallurgical products. J. Kinnunen and B. Wennerstrand (*Chemist Analyst*, 1954, **43** [4], 88–89).—A 0·5 to 5-g sample is dissolved in conc. HNO_3, diluted with an equal vol. of H_2O, 3 to 5 g of tartaric acid are added and the solution is neutralised with conc. ammonia, 5 to 10 ml being added in excess. The blue colour of Cu·· is discharged by adding 20 per cent. KCN solution; 5 ml are added in excess; 0·1 M ethylenediaminetetra-acetic acid solution (I) is added in excess to mask any Pb. Two ml of 2 per

cent. diethyldithiocarbamate (II) are added, and the solution is extracted with chloroform; the extract is diluted to 25 ml and the optical density is measured at 420 mμ. If too much Bi is present for this procedure, enough II is added to ppt. the Bi com-pletely; after extracting with chloroform, 10 ml of dil. nitric acid (1 + 1) are added to the extract, which is then evaporated down to 3 ml.; 2 g of am-monium acetate are added, the solution is diluted to 150 ml, a few drops of 0·1 per cent. catechol violet are added and then conc. ammonia until the solution turns blue. The solution is titrated with 0·01 M I until the colour turns yellow when viewed by transmitted blue light. Samples that have a low bismuth content are dissolved in nitric acid and pptd. with HgO from a boiling, slightly acid solution; after being washed, the ppt. is dissolved in nitric acid and treated as above. Catechol violet may be replaced by KI at pH 3 to 4, by thiourea at pH 1·1 to 2·3, or by thio-cyanate at pH 0·5 to 1·5.   H. P. PAGET

1195. Extension of isotopic method for deter-mining oxygen in metals to copper containing 0·01 to 0·1 weight per cent. of oxygen. A. D. Kirshen-baum and A. V. Grosse (*Anal. Chem.*, 1954, **26** [12], 1955–1956).—A sample of Cu metal (8 to 10 g) is mixed with a known wt. of $Cu^{18}O$ "master alloy" ($\simeq$ 100 mg) that has been prepared by heating Cu containing 0·579 per cent. of O in an atmosphere of ^{18}O at 400° C. The mixture is heated in a molybdenum, platinum or carbon crucible in the presence of C ($\simeq$ 10 mg) of known C content under $<10^{-4}$ mm at 1100° to 1200°C (induction furnace). The liberated CO is collected and, from the mass-spectrometrically determined ^{18}O to ^{16}O ratio, the O content of the sample is deduced. Results for four samples are quoted.   D. A. PANTONY

1196. Determination of oxygen in zinc, cadmium and lead. H. Hartmann and G. Ströhl (*Z. anal. Chem.*, 1955, **144** [5], 332–342).—A method is described for determining small quantities of oxygen in metallic zinc, cadmium, lead and other low-melting metals. The water liberated, *e.g.*, accord-ing to the equation $ZnO + H_2S \rightarrow ZnS + H_2O$, is determined gravimetrically. The metal sample, which is thoroughly cleaned to remove surface oxides, grease, water, etc., is placed in a special furnace, in which the air and water are displaced with purified and dried nitrogen. The furnace is heated until the metal melts, and then purified and dried H_2S is passed in; any H_2O formed being collected in absorption tubes containing P_2O_5. After 30 min., any H_2O vapour still in the furnace is displaced by a further stream of nitrogen and the water is weighed. Typical samples of Zn contained 1 to 3 g of O per ton; Cd, about 3·5 g per ton; and Pb, about 7 g per ton. The determination takes about 2 hr. and the error is ± 5 per cent.
                          P. S. STROSS

1197. Determination of superoxide oxygen [in peroxides] with chlorine dioxide C. B. Riolo (*Ann. Chim., Roma*, 1954, **44** [10], 815–820).—A solution of ClO_2 in CCl_4 converts superoxides quantitatively into peroxides: $2KO_2 + 2ClO_2 \rightarrow Cl_2 + K_2O_2 + 3O_2$. A weight of sample containing 0·04 g of superoxide in 5 ml of CCl_4 is treated with 2 ml of CCl_4 containing slightly more than the stoicheiometric equivalent of ClO_2 for $\simeq$ 30 min. at 0°C. The O is measured in a gas burette. Samples of commercial Na_2O_2 contained $\simeq$ 0·5 per cent. of NaO_2; CaO_2 prepared

by Traube and Schulze's method contained $\simeq 8\cdot7$ per cent. of superoxide.　　　T. P. McLaughlin

1198. Determination of total sulphur in coal and coke by the Eschka method. I. Precipitation and recovery of soluble sulphates as barium sulphate. R. A. Mott, D. A. Ruell and H. C. Wilkinson (*Fuel, Lond.*, 1955, **34** [1], 78–86; 87–94).—*Procedure*— Ten ml of a 10 per cent. solution of $BaCl_2.2H_2O$ are quickly added to an acid solution of the sulphates which has been boiled to remove CO_2 and which has a pH of $1\cdot5$ to $0\cdot5$, and the mixture is shaken. The concentration of $Ba^{..}$ is sufficient to counteract any tendency to solvation of sulphate by the excess of acid and to give crystals of $BaSO_4$, which can be filtered off immediately. Recovery should be $100 \pm 1\cdot5$ per cent. of the theoretical. The time of settling is decreased. **II. Conversion of the sulphur in coal and coke into soluble sulphates and determination of the blank.** The time of heating of the sample with Eschka mixture is reduced from $2\cdot5$ hr. by: (*a*) heating to 775° C during 1 hr. and continuing for 0·5 hr.; (*b*) heating for 20 min. in the cool zone of a zoned muffle and for 1 hr. in the hot zone at 775° C. Coke or coal containing 22·5 per cent. of volatile matter may be plunged directly into the muffle at 775° C on a 6-mm silica plate and heated for 90 min. and 60 min., respectively. Loss of S is minimised. It is desirable to conduct a blank experiment using the full procedure and to add sulphur equivalent to 8 mg of $BaSO_4$ to the solution before pptn.　　　J. G. King

1199. Sulphur in coal. III. Determination of the total sulphur in its separate forms. V. Gómez Aranda and J. Auría Arbuniés (*Combustibles*, 1954, **14**, 171–180).—Sulphate S and non-pyrites Fe are easily extracted from coal by boiling for 30 min. with 5 N HCl. Pyrites and marcasite are completely dissolved by boiling with 4 N HNO_3 at ordinary temp. for 48 hr. or with 2 N HNO_3 by boiling for 30 min. The organic sulphur is partly attacked by this treatment with solution of the products; the pyrites S is therefore determined by multiplying the pyrites Fe by 1·148. In lower category coal, the pyrites Fe determination is difficult because of the coloured organic compounds extracted by the HNO_3; here, the total Fe is determined from the ash, and the non-pyrites Fe subtracted from this figure. The pyrites and marcasite occurring in Aragonese coal correspond to the formula FeS_2. *Procedure*—About 5 g of coal, weighed accurately, are boiled with 100 ml of 5 N HCl for 30 min., filtered off and washed seven times with dil. $(1 + 20)$ HCl. The filtrate is treated with Br water to oxidise Fe^{II} to Fe^{III}, and the Fe is pptd. with aq. NH_3. The ppt. is filtered off and washed four or five times with hot water; it can be used for the determination of the non-pyrites Fe, while the filtrate and washings are combined and the sulphate content is obtained as $BaSO_4$ (*cf.* Gómez Aranda and Auria Arbuniés, *Anal. Abstr.*, 1954, **1**, 2668). The oxidation method for pyrites S is a slight variation of the method of Powell and Parr (*Univ. Ill., Bull.*, 1919, [111], 62) or of that due to Mott (*Brit. Abstr.*, C, 1950, 360). Organic S = total S − (sulphate S + pyrites S). For the determination of pyrites Fe, 1 g of the carbonaceous residue, after extraction of sulphate S and non-pyrites Fe, is boiled for 30 min. with 50 ml of 2 N HNO_3, filtered off and the residue washed six times with 2 N HNO_3, using a total of 20 ml. The Fe is pptd. with 3 N aq. NH_3, filtered off, dissolved in 6 N HCl and estimated with 0·02 N $K_2Cr_2O_7$.　　　D. Leighton

1200. Determination of the separate forms of sulphur in lower quality coals. Method adopted by the Saragossa section of the Instituto Nacional del Combustible (*Combustibles*, 1954, **14** [74], 181–182).—For the determination of sulphate S, 5 g of the sample, passing through a 0·2-mm-aperture screen, are boiled with 100 ml of 5 N HCl for 30 min., filtered off and washed with 20 ml of dil. HCl $(1 + 19)$. The filtrate is treated with 1 ml of Br water and the Fe is then pptd. with aq. NH_3, washed four or five times with hot water and dissolved in 10 ml of hot 6 N HCl, the filter is washed with another 10 ml of 6 N HCl and with hot water until completely white. The solution is kept for the determination of non-pyrites Fe. The filtrate from the Fe pptn. is analysed for sulphate S by the normal $BaSO_4$ pptn. method. For the determination of pyrites S, 1 g of the sample is ashed and the ash treated with 50 ml of boiling 6 N HCl, until the residue is a white powder. The volume is reduced to about 20 ml and a solution of $SnCl_2$ (5 g in 50 ml of HCl, then addition of 50 ml of water) is added dropwise with agitation until the yellow colour of the Fe^{III} ion disappears, when five more drops are added. To the product, 10 ml of 6 per cent. aq. $HgCl_2$ solution are added, giving a ppt. of Hg_2Cl_2, followed by 15 ml of a $H_2SO_4 - H_3PO_4$ mixture [150 ml of H_2SO_4 (d 1·84) plus 150 ml of H_3PO_4 (d 1·75) diluted $1 + 1$]. The whole is diluted to 150 to 200 ml and titrated with 0·0179 N $K_2Cr_2O_7$, using 0·2 per cent. aq. Na diphenylaminesulphonate as indicator. A conversion factor is used to calculate pyrites sulphur. Organic S is determined by difference from total S.　　　D. Leighton

1201. Determination of total sulphur in solid fuels. V. Gómez Aranda and J. Auría Arbuniés (*Combustibles*, 1954, **14**, 124–125).—The authors' method (*Anal. Abstr.*, 1954, **1**, 2668), modified by the extraction with 150 ml of boiling water for 20 to 25 min. followed twice by 50 ml for 5 to 10 min. each, instead of the former once with 200 ml for 30 min., is compared with those of Rodriguez Pire and Corrales Zarauza (*Inst. Nac. Carbon Bol. Inf.*, 1954, [3]) and Strambi (*Il Calore*, 1954, [3]). It is concluded that whilst it is convenient to have two or more methods of equal validity, the Eschka method is most suitable for laboratories carrying out numerous assays.　　　D. Leighton

1202. Modification of the Willard - Schneidewind determination of sulphate in chroming baths. J. Ibarz Aznárez and J. B. Vericat Raga (*An. Soc. Esp. Fis. Quim.*, B, 1954, **50** [12], 989–990).—The accuracy of the Willard - Schneidewind method (*Trans. Amer. Electrochem. Soc.*, 1929, **56**) is increased by raising the concn. of acetic acid in the pptn. medium to 50 per cent., to avoid the errors caused on slow pptn. of $BaSO_4$, and by washing the ppt. with 2 per cent. acetic acid to remove the occluded chromic salts.　　　D. Leighton

1203. Oxidation with alkaline permanganate using formic acid for the back titration. II. Potentiometric determination of chromium. I. M. Issa, A. A. Abdul Azim and R. M. Issa (*Anal. Chim. Acta*, 1955, **12** [1], 92–100).—Titrations of $Cr^{...}$ with $KMnO_4$ fail to give accurate results. A soln. of $KMnO_4$ (0·126 N) can be titrated with a $Cr^{...}$ soln. (0·04 to 0·18 N), if the $KMnO_4$ soln. is made 0·8 to 1·5 N with respect to NaOH and the titration is carried out in the presence of $Ba^{..}$, equivalent to, or 50 per cent. in excess of, the MnO_4'' and CrO_4'' formed in the reaction. Alternatively, a

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$Cr^{...}$ soln. is added to an excess of $KMnO_4$ soln. (0·12 N), made 2·5 N in respect of NaOH in the absence of $Ba^{..}$ or N in NaOH if $Ba^{..}$ is present, and the excess of $KMnO_4$ is titrated with formic acid soln. (0·016 to 0·6 N) or, in the presence of $Ba^{..}$, with $Pb^{..}$ soln. (0·1 N).   W. C. JOHNSON

1204. Function of silver ions in the oxidation of chromium (III) to chromium (VI) or manganese (II) to manganese (VII) in acid solution by ammonium persulphate. W. Oelschläger (Z. anal. Chem., 1955, **144** [1], 27–28).—The rate of oxidation of Cr^{III} (and Mn^{II}) by $(NH_4)_2S_2O_8$ in 0·2 N H_2SO_4 is considerably increased by the addition of $Ag^{.}$; transitory formation of ozone is then probable.   D. R. GLASSON

1205. [Room temperature ductility of chromium.] Appendix. Analytical methods used for determination of nitrogen and oxygen in chromium. E. J. Lumley (J. Inst. Metals, 1954, **83** [4], 141).—A modified Allen method is used for the determination of nitrogen. The pulverised sample (0·5 g) is dissolved in dil. H_2SO_4, a little $BaCl_2$ is added, and then insol. nitride is centrifuged off with the $BaSO_4$ ppt. The ppt. is decomposed by boiling with H_2SO_4 and K_2SO_4 and the NH_3 formed is distilled in a semi-micro unit. The distillate is nesslerised and the colour is measured by means of a Spekker absorptiometer (Ilford filter No. 601). A blank determination is necessary. By this method, 2×10^{-6} g of nitrogen can be estimated with good reproducibility. Oxygen is determined as follows: the specimen is heated for 1 hr. at 850° C in vacuo to convert the oxygen to Cr_2O_3. A 1-g sample is then dissolved in hot 10 per cent. aq. HCl, cooled for 2 hr. and the ppt. of Cr_2O_3 is filtered off, washed with 4 per cent. aq. HCl, ignited and weighed.   S.C.I. ABSTR.

1206. Semi-quantitative analysis of elemental selenium. J. W. Mellichamp (Applied Spectroscopy, 1954, **8** [3], 114–117).—A 50-mg sample in the "as received" condition is excited in the 10-amp. d.c. arc and burnt to completion. The background of the spectrogram is used as the internal standard. Calibration is effected by the use of synthetic standards containing 24 possible impurity elements in the range 0·1 p.p.m. to 1000 p.p.m. in steps of ten parts per million.   B. S. COOPER

1207. Investigations on the applicability of di-ethyldithiocarbamate in analysis. III. Photometric determination of tellurium and its separation from large amounts of selenium. H. Bode (Z. anal. Chem., 1955, **144** [2], 90–100).—Te is separable from Se by extraction with diethyldithiocarbamate and CCl_4 at pH 8·5 to 8·7. Measurement of the absorption of the extracts at 428 mμ affords a quant. photometric determination of Te, even for ratios of Te to Se of 0·5 p.p.m.   D. R. GLASSON

1208. Critical study of the gravimetric determination of tungsten by means of benzidine. J. Zato (An. Soc. Esp. Fís. Quím., B, 1954, **50** [12], 977–980).—Pptn. of ortho-, meta- and para-tungstates from pure solution by benzidine is quantitative; this is not affected by the presence of alkali sulphides or K_2SiO_3, up to 60 per cent. of the amount of WO_3 present, when pptn. is at increased temp. Silicic acid present when pptn. is at room temp. causes high results. Results were as much as 4 per cent. low in the presence of up to 20 per cent. of NaCl. The method of Knorr (F. Welcher, "Organic Analytical Reagents," New York, 1948, Vol. II, p. 312) is thus not applicable to analysis of tungsten ores which have to be decomposed by alkali and the extract neutralised with HCl.   D. LEIGHTON

1209. An improvement in the determination of uranium-235 by radioactivation. A. P. Seyfang (Analyst, 1955, **80**, 74–76).—The precision of the method previously described (Seyfang et al., Brit. Abstr. C, 1953, 421) for the determination of ^{235}U in mixtures of naturally occurring U isotopes by radio-activation and measurement of the fission product ^{140}Ba has been considerably increased, mainly by paying stricter attention to the counting equipment and to the technique used for counting. With the equipment described, the precision is five times as good as that previously reported.   A. O. JONES

1210. Volumetric determination of uranium in poor ores. W. M. Smit and J. Klinkhamer (Rec. Trav. Chim. Pays-Bas, 1954, **73** [11], 1009–1021).—Potential - pH diagrams for aq. solutions of $Fe^{..}$/ $Fe^{...}$ and for aq. solutions of $U^{...}$, $U^{....}$ and $UO_2^{..}$ are developed for molar solutions of the ions concerned, and the relevant part of the uranium diagram is recalculated for the limits of concentration to be found in the required titration. Fe and U in solution are reduced in a Jones reductor to $Fe^{..}$, $U^{...}$ and $U^{....}$. As the oxidation of $U^{...}$ to $U^{....}$ is reversible on a smooth platinum electrode and $U^{....}$ to $UO_2^{..}$ is not, the solution can be titrated with $KMnO_4$ by a dead-stop technique. Hence, under an applied potential of 100 mV, the current falls until all the $U^{...}$ is oxidised to $U^{....}$, when it remains zero until all the $U^{....}$ is oxidised to $UO_2^{..}$, and then rises suddenly on the start of the oxidation of $Fe^{..}$ to $Fe^{...}$. Suitable pH is necessary (0·75 to 1·7) in order that the $UO_2^{..}$ does not appreciably oxidise the $Fe^{..}$.   E. J. H. BIRCH

1211. The potentiometric reduction of uranyl chloride solutions. H. K. El-Shamy and S. El-Din Zayan (Analyst, 1955, **80**, 65–68).—An examination of the conditions for the potentiometric reduction of uranyl chloride soln. shows that U can be accurately determined potentiometrically by direct reduction to the quadrivalent state with $CrCl_2$ soln. Equilibria are attained more rapidly and the inflexions in the graph indicating complete reduction are steeper at higher temp. than at room temp. The fall of potential near the equivalence point increases with acidity and reaches a max. value in 8 M HCl. In the presence of 8 M HCl and complexing acids, particularly tartaric acid, equilibria are attained instantaneously. Addition of KCl is still more effective and improves the titration graph even with 0·1 M HCl at room temp.   A. O. JONES

1212. Determination of hydrofluoric acid in nitric - hydrofluoric acid mixtures. S. A. Long (Anal. Chem., 1954, **26** [12], 1988–1989).—The sample soln. (5 ml) is added to 0·375 per cent. H_2O_2 (40 ml). A conc. soln. of NaOH (6·5 g) is added, followed by 0·5 N NaOH until the soln. is alkaline (phenolphthalein). NaCl (30 g) is added, followed by 0·5 N HCl until the soln. is just acid. At 70° to 80° C, the soln. is titrated with 0·01 N NaOH, and then with 0·16 M $Al^{...}$ (as chloride) that has been standardised against NaF (methyl red). After the determination of the sp.gr. of the soln., the HF concn. is calculated from a given formula. Mean deviation of thirty results over the range 0·03 to 0·62 per cent. of HF is given as 0·005 per cent.   D. A. PANTONY

1213. Spectrophotometric determination of fluorides in slags, fluxes and other materials. Application of a method devised by the Food Research Laboratories of New York. L. Bloch (Chem. Weekbl., 1955,

51 [5], 65–67).—In this method, published by Icken and Blank (*Anal. Abstr.*, 1954, **1**, 695), F is separated from the sample as H_2SiF_6 by distillation of the sample with $HClO_4$ in a current of steam. The distillate is neutralised to *p*-nitrophenol by the addition of 0·05 *N* KOH and the F is determined by the bleaching effect on a Th - alizarin lake. The transmittance is measured with a spectrophotometer at 525 mμ. A solution containing 10 μg of F per ml is used as a blank. A. J. Mee

1214. Determination of chloride and bromide in halide mixtures. C. Mahr and H. Otterbein (*Angew. Chem.*, 1954, **66** [20], 636–638).—By oxidising a bromide - chloride solution of appropriate acidity with $Ce(SO_4)_2$, the Cl′ is left intact and the Br′ is oxidised to free bromine. In an example, 30 per cent. H_2SO_4 is added to a halide solution containing < 30 mg of Cl′ and 100 mg of total halide such that the solution has an acidity of 1 to 2·5 *N*. Not more than 30 ml of this solution are used per determination. For every 35 mg of Br′, 0·2 to 0·3 g of analytically pure $Ce(SO_4)_2$ is added. The Br liberated is steam-distilled into a solution containing 10 ml of 3 per cent. H_2O_2 and 10 ml of 5 per cent. NaOH for 5 to 7 min. and determined potentiometrically. The solution left in the reaction flask contains the Cl′ and is treated with H_2O_2 in a dropwise manner in the presence of 2 ml of 60 per cent. H_2SO_4 until the $Ce(SO_4)_2$ is reduced; this solution is then titrated against $AgNO_3$. The method is applicable, with suitable modifications, to the potentiometric determination of Cl′ and Br′ mixed with I′, CN′ and CNS′. S.C.I. Abstr.

1215. Method for sparking thin-sheet samples for spectrographic analysis; application to manganese and niobium determination in stainless steel. F. P. Landis and L. P. Pepkowitz (*Anal. Chem.*, 1955, **27** [1], 141–142).—Excessive volatilisation of Mn and Nb during their spectrographic determination in steel is prevented by passing a stream of helium at 0° C over the upper unsparked side of the steel sheet. The rate of flow of He is from 5 to 30 litres per min. according to the thickness of the sheet. The precision of the method (useful for identifying large stocks of steel sheet) equals that obtained with mass samples, but the values obtained must be corrected by a chemical analysis of one piece of steel of each thickness. W. J. Baker

1216. Determination of manganese in copper and ferrous alloys. J. Kinnunen and B. Merikanto (*Chemist Analyst*, 1954, **43** [4], 93–95).—The sample is dissolved in dil. aq. nitric acid (1 + 1), and nitric oxides are removed by boiling with added sulphamic acid; Cu is then determined iodimetrically. Solid ascorbic acid is added first, then 3 ml of 20 per cent. potassium cyanide for each 0·1 g of sample and finally 10 ml of conc. aq. NH_3. The solution is titrated with 0·1 *M* ethylenediaminetetra-acetic acid solution (**I**), by means of Erichrome black T indicator; for alloys rich in Mn, the solution should be hot. If Al is present, it should be masked by treating with ammonium fluoride. If Pb is present, it may be pptd. by adding Na diethyldithiocarbamate (**II**) before the indicator is added; alternatively the Pb and Mn may be titrated jointly with **I**; **II** is added and the liberated **I** (equivalent to the Pb) titrated with a standard Mn solution. Steel samples are dissolved in conc. hydrochloric acid and the Fe is extracted with pentyl acetate; after dilution, ascorbic acid, ammonia and cyanide are added, and the Mn is titrated with 0·01 *M* **I**. Alternatively,

the dissolved sample is treated with ammonium bifluoride to mask the Fe, ammonium thiocyanate is added and the solution is extracted with butyl phosphate - ether (3 + 2). The solvent is treated with conc. hydrochloric acid, water is added and the aqueous phase is separated; after addition of ascorbic acid, ammonia and cyanide, the solution is titrated with **I** as before. H. P. Paget

1217. Analytical separation of rhenium and molybdenum by ion exchange. V. W. Meloche and A. F. Preuss (*Anal. Chem.*, 1954, **26** [12], 1911–1914).—A method for the anion-exchange separation of Mo and Re that is claimed to save time and reagents is examined. *Procedure.*—The flue dust containing Mo and Re (1 to 1·5 g) is dissolved by fusion in NaOH - Na_2O_2 (see *Brit. Abstr. C*, 1952, 1087) and then leached with hot water. The soln. is cooled to 5° C and filtered; the filtrate is passed through an ion-exchange resin [Amberlite IRA-400 (5 g) in perchlorate form] at 10 to 20 ml per min. Water (100 ml) at 1 to 2 ml per min. is used for elution. The adsorbed Mo^{VI} is reduced, and hence eluted, by *M* K oxalate (300 ml), followed by water (50 ml), and the Re^{VI} is subsequently removed from the column by washing with *M* $HClO_4$ (200 ml). Mo and Re are determined by standard methods. Results are given for synthetic mixtures containing Mo to Re ratios of 10^2, 10^3 and 10^4 to 1. SeO_3'' and SeO_4'' are not adsorbed on the column, and AsO_4''' behaves similarly to MoO_4''. D. A. Pantony

1218. Colorimetric determination of iron. M. Grat-Cabanac (*Anal. Chim. Acta*, 1955, **12** [1], 50–53).—The colorimetric methods for Fe determination with thiocyanate or 2 : 2′-dipyridyl are modified by the addition of methylene blue or indigo carmine. A range of tints from blue to red is produced and the intermediate violet colour is highly sensitive to variations in Fe content. With CNS′, this sensitivity corresponds to 0·05 μg of Fe per ml, and with 2 : 2′-dipyridyl, 0·01 μg of Fe per ml. To determine Fe with CNS′, a suitable vol. of the soln. is treated with 0·5 ml of conc. HCl (sp. gr. 1·18), 4 ml of 20 per cent. aq. NH_4CNS and 0·5 ml of aq. H_2O_2 (20 vol.). A measured vol. of 0·05 per cent. aq. indigo sulphate is then added to produce a suitable violet tint. The same vol. of aq. indigo carmine is then added to a mixture of the other reagents and aq. $FeCl_3$ (1 ml $\equiv$ 10 μg of Fe) is added to produce the same tint as that of the test. The vol. of aq. $FeCl_3$ required indicates the quantity of Fe··· in the test. A similar procedure is used with 2 : 2′-dipyridyl; if dithionite is used to reduce the Fe, the excess is destroyed by boiling, and hydroxylamine hydrochloride is added. W. C. Johnson

1219. Separation of iron from cerium by partition between two solution media. H. Specker and M. Kuchtner (*Z. anal. Chem.*, 1955, **144** [1], 25–27).—Fe and Ce can be quant. separated in the presence of NH_4CNS ($\simeq$ 2·5 *N*) in HCl ($\simeq$ 3 *N*) solution by shaking with ether - tetrahydrofuran mixtures; three (often two) extractions within 10 min. are sufficient for separating amounts of $\simeq$ 10 to 150 mg of Fe and Ce. D. R. Glasson

1220. Determination of a small amount of ferric ion and the reduction of uranium in the presence of air with the aid of liquid amalgam. C. Yoshimura (*J. Chem. Soc. Japan*, 1954, **75** [6], 603–605).—Generally, Fe··· can be readily reduced to Fe·· with liquid zinc amalgam, but when the amount of

Fe is < 2 mg, the formation of traces of H_2O_2, which are produced from dissolved O and nascent H in the soln., can cause an uncontrollable error, even when the determination is carried out in a current of CO_2. To eliminate this defect, reduction of Fe with bismuth amalgam in 6 to 10 N H_2SO_4 is desirable. To remove suspended micro-particles of the amalgam after the reduction, the soln. is shaken with a small amount of CCl_4. When Fe is reduced with Zn - Hg in 1 to 2 N H_2SO_4 by the usual method, $\simeq 0.3$ mg of H_2O_2 is produced (determined with diphenylcarbazide), whilst 0·01 to 0·004 mg of H_2O_2 is produced when Bi - Hg in 6 to 10 N H_2SO_4 is used. Bi - Hg can also be used for the reduction of U‥‥‥ to U‥‥.   K. SAITO

1221. The compleximetric determination of iron by use of Chromazurol S as indicator. A. Musil and M. Theis (*Z. anal. Chem.*, 1955, **144** [5], 351–353).—Geigy Chromazurol S (**I**) is a suitable reversible indicator for the titration of Fe. Al, like Mn and Ti, does not interfere but must be determined in a separate aliquot. *Procedure*—Adjust the pH of the solution to 2, heat to 60° C and add 2 M sodium acetate (3 ml) and a little chloroacetic acid. Add 0·4 per cent. aq. **I** (5 drops) and titrate with 0·05 M complexone III to a bright orange colour.   P. S. STROSS

1222. Determination of free acid in ferric solutions. J. A. Bishop and S. Summ (*Chemist Analyst*, 1954, **43** [4], 96–97).—Titration of free acid in solutions containing Fe‥‥ is difficult owing to the formation of coloured solutions or ppt. when complexing agents such as tartrate, salicylate, ethylenediaminetetra-acetate, oxalate or fluoride are added. Citrate gives no ppt. and the green colour of the solution is in contrast to the colour of phenolphthalein. *Procedure*—The sample solution is saturated with Na or K citrate, 10 drops of 1 per cent. phenolphthalein are added and the titration is carried out with NaOH, standardised against K hydrogen phthalate. The solution must be nearly saturated with citrate at the end-point. The free-acid content of the sample is equivalent to the amount of base used.   H. P. PAGET

1223. A new qualitative identification procedure for cobaltous and nickelous ions. S. K. Yasuda and J. L. Lambert (*J. Chem. Educ.*, 1954, **31** [11], 572).—A method is given for the identification of Co‥ and Ni‥ by the use of monothiophosphate ion, PO_3S'''. Co‥ forms a deep-blue solution with an excess of PO_3S''' in a solution made slightly alkaline with sodium acetate. Under the same conditions, Ni‥ forms a greenish-white precipitate, which does not interfere with the formation of the Co compound. Nickel and cobalt are separated as sulphides by normal group analysis, the mixture of sulphides being dissolved in aqua regia and the solution evaporated almost to dryness before dilution to 10 to 15 ml. Sodium acetate (1 g) is then added, and the solution is boiled for one minute, filtered and cooled to room temperature. To one half of the filtrate is added, with vigorous shaking, 0·1 to 0·2 g of anhydrous Na_3PO_3S. A deep-blue solution is formed by Co‥. Dimethylglyoxime in alcoholic solution is added to the other portion to identify nickel.   A. LEDWITH

1224. Colorimetric determination of cobalt with $2':2':2''$-terpyridine [$2:2':2''$-terpyridyl]. R. R. Miller and W. W. Brandt (*Anal. Chem.*, 1954, **26** [12], 1968–1969).—The factors influencing the quant. production of the Co‥ - terpyridyl colour are examined. *Procedure*—A soln. of Co^{II} is adjusted to 1 to 4 mg per 100 ml. To a 25-ml aliquot is added $HClO_4$ (1 + 1) (1 drop) and the pH is adjusted to 2 to 11 (6 N NaOH or 6 N HCl). The mixture is diluted to 50 ml and the reagent [0·1 per cent. $2:2':2''$-terpyridyl in dil. HCl (3 to 5 ml)] is added; the colour is extracted into nitrobenzene (20 ml) and, after filtration through glass wool, the absorption of the organic phase is measured at 510 mμ. Effects of twenty other ions are listed and of these, Fe‥‥, Fe‥ and VO_3' and oxidising agents interfere seriously.   D. A. PANTONY

1225. Separation of cobalt and zinc by liquid - liquid extraction. H. A. Mahlman, G. W. Leddicotte and F. L. Moore (*Anal Chem.*, 1954, **26** [12], 1939–1941).—A 3 N HCl soln. of Co‥ and Zn‥ (1 vol.) is mechanically shaken with an 8 per cent. w/v soln. of methyldioctylamine in trichloroethylene. After separation of the aq. phase, the organic layer is washed with 3 N HCl, and the aq. phases are combined. From the org. phase, Zn‥ is recovered by extraction with 0·05 N HCl and is determined by a standard mercurithiocyanate gravimetric method. Co‥, Ni‥, Cr‥‥ and Mn‥ remain in the aq. phase, but Fe‥‥ and Cu‥ are distributed between the solvents and must be removed by preliminary treatment. The quantitative separation of Zn‥ is followed by a radio-tracer technique, and the method is claimed to be adaptable to the determination of Zn‥ by an isotopic dilution method.
                       D. A. PANTONY

1226. High-frequency titrations involving chelation with ethylenediaminetetra-acetic acid. II. Quantitative determination of some divalent metals. R. Hara and P. W. West (*Anal. Chim. Acta*, 1955, **12** [1], 72–78).—The Sargent Model V oscillometer is applied to the titration of various metals with the use of ethylenediaminetetra-acetic acid (**I**). Nickel (0·4 to 1·4 mg) is added to 1/3480 M **I** (100 ml) and the liberated H⁺ are titrated with 1/87 M NaOH. Direct titrations of Co‥, Ni‥, Cu‥, Zn‥, Cd‥, Pb‥ and Mn‥ in 0·001 to 0·000167 M concn. with the disodium salt of **I**, preferably in the presence of an acetate buffer of pH 5·3 (1 ml per 100 ml of soln.), yield curves with clear end-point inflexions. Calcium is best titrated in the presence of a phosphate buffer of pH 7, but Ba, Sr and Mg give erratic results. Large concn. of electrolytes disturb these titrations.   W. C. JOHNSON

1227. The determination of nickel with dimethylglyoxime in the presence of iron and cobalt. E. E. Byrn and J. H. Robertson (*Anal. Chim. Acta*, 1955, **12** [1], 34–37).—When Ni is pptd. with dimethylglyoxime (**I**) in the presence of Fe‥‥ and Co‥, the ppt. is contaminated with a complex containing Fe and Co (*Brit. Abstr. C*, 1948, 5). The co-precipitation of these metals is prevented by the addition of NN-di(hydroxyethyl)glycine (**II**). Nickel (34 to 68 mg) is separated quant. from Co (up to 14·7 mg) and Fe (up to 825 mg) by the usual pptn. with **I**, after the addition of 1 to 12·5 g of **II**.
                       W. C. JOHNSON

1228. A spectrochemical method for the analysis of M-252 nickel-base high-temperature alloy and the preparation of standards by powder metallurgy. W. O. Gerber, jun., and W. H. Tobin (*Applied Spectroscopy*, 1954, **8** [3], 120–125).—The M-252 nickel-base high-temperature alloy contains nine elements in addition to nickel. Of these, carbon is determined by a high-frequency induction furnace, Mo, Co, Fe and Cr by X-ray fluorescence, whilst Ti, Al,

Mn and Si are best determined by emission spectroscopy. Calibration of the spectrographic procedures may be effected by the use of a series of chemically analysed samples, but Al and Ti in particular are difficult to determine accurately by chemical methods. A satisfactory series of calibration standards has been prepared by powder metallurgy. B. S. COOPER

1229. Spectrophotometric determination of palladium with 2-nitroso-1-naphthol. Kuang Lu Cheng (*Anal. Chem.*, 1954, **26** [12], 1894–1895).—Factors influencing the quant. production of the violet or orange-coloured ppt. of $PdCl_4''$ with 2-nitroso-1-naphthol, and its extraction into benzene or toluene, are examined. Standards were prepared from $PdCl_2$ or Pd (0·1 mg of Pd per ml of stock soln. diluted to 5 μg per ml), and suitable aliquots are treated with 3 N HCl (2 drops), 3 per cent. disodium ethylenediaminetetra-acetate (1 ml) and 1 per cent. 2-nitroso-1-naphthol in 95 per cent. aq. ethanol (0·1 ml). After 10 min., toluene (5 ml) and 14 per cent. aq. NH_3 (1 ml) are added and the complex is extracted into the organic phase; the colour is measured spectrophotometrically (370 mμ), after centrifugation. The Beer - Lambert law holds over the range 5 to 25 μg of Pd per 5 ml of toluene. The determination of Pd in an unknown soln. is similar, the initial pH of the soln. having been adjusted to 2·5. The procedure gives an accuracy of $<$ 1 per cent., and the limit of detection is 5 p.p.m. $Fe^{...}$, $Co^{..}$, $Ni^{..}$, $Cu^{..}$, $Cr^{...}$, $AuCl_4'$, $Zr^{....}$ and the Pt metals do not interfere, but CN' destroys the complex. D. A. PANTONY

1230. The separation of the tri-, tetra- and hexavalent states of plutonium by paper chromatography. M. R. Cowan and J. K. Foreman (*Chem. & Ind.*, 1954, [52], 1583–1585).—Experimental details are given of the quantitative chromatographic separations of $Pu^{....}$ and $Pu^{......}$ with either *n*-butanol saturated with 3 N HCl or *n*-pentanol saturated with 4 N HCl as developing solvent. $Pu^{...}$ and $Pu^{....}$ are separated by means of miscible mixtures of ethyl acetate and HCl. C. H. WHITTON

1231. Standard samples of coal for the estimation of arsenic content. Institute of Brewing Analysis Committee (*J. Inst. Brewing*, 1954, **60** [6], 462–463). —The methods of analysis of coal for arsenic content are reviewed. The Marsh method is satisfactory in experienced hands; the Case method (*J. Inst. Brewing*, 1938, **44**, 362) and the British Standard Method (Methods for the Analysis and Testing of Coal and Coke, B.S. 1016, pp. 50 to 53) are recommended. Attention is drawn to the following points: sampling, purity of reagents and control of humidity of evolved gases in the Gutzeit method; this last may be accomplished by the use of glycerol in the lead acetate solution. Loss of arsenic during ignition should be avoided by thoroughly moistening the sample with lime-water. A standard sample of coal having an arsenic content of 9·0 $\pm$ 0·1 p.p.m. As_2O_3 (average of 212 estimations carried out in 9 laboratories) is available from the Institute of Brewing. The standard error for a single determination is $\pm$ 1·8 p.p.m. and for the average of duplicates $\pm$ 1·3 p.p.m. G. B. THACKRAY

1232. Elementary analysis of coal, especially the direct oxygen determination. H. W. Deinum (*Brennstoff-Chem.*, 1954, **35** [21–22], 334–337).— The system of estimating the moisture, ash, C, H, N, S and O described is suitable for coal containing $<$ 2 per cent. of ash. For all estimations, a weighed 100-mg finely ground sample of 0·3-mm particle size is used. Moisture is determined by passing a stream of oxygen-free nitrogen over the sample heated to 105° C and absorbing the moisture from the stream in a drying medium [magnesium perchlorate (**I**)]. Carbon and hydrogen are determined by combustion to CO_2 and H_2O, by passing electrolytic O [after being purified by passage over CuO at 750° C and through soda - asbestos (**II**) and **I**] over the electrically heated sample, the combustion products being passed successively over CuO heated to 800° C, heated Ag (to bind halogen) and $PbCrO_4$ heated to 600° C (to bind SO_2). They are then passed successively through **I** to absorb the water of combustion, through $K_2Cr_2O_7$ and H_2SO_4 on silica gel to remove oxides of N and finally through **II** to absorb the CO_2 of combustion. Ash is determined by weighing the sample after combustion. The N is determined by an improved Dumas method. The sample is burnt in a stream of mixture of O and CO_2, and N is collected. Excess of the oxidising gas is passed over the electrically heated sample, the excess of oxygen being removed from the reaction gases by Ni and Cu at 1000° C. The reaction gases then pass first over CuO at 550° C (to oxidise any CO to CO_2) and then through 50 per cent. aq. KOH to remove CO_2, the remaining purified N being collected in a nitrometer. The Kjeldahl method of determining N is found to be too slow and to introduce too high an error. The S is determined by the Eschka method (sulphate determination) by burning the sample and estimating the resulting SO_2, or oxidising this (with H_2O_2) to H_2SO_4 and titrating, or (for younger coals or those of small sulphur content) oxidising the SO_2 to SO_3, fixing the SO_3 on metallic silver and estimating the Ag_2SO_4. For the direct estimation of O, a stream of oxygen-free nitrogen (produced by passing technical N over heated Cu and then through **I** and **II**) is led through a quartz combustion tube containing the sample heated to 1120° C, the reaction products are passed over ash-free C heated to 1120° C and the CO produced is estimated. In the method described, the reaction products are oxidised to CO_2 by passage over red HgO at 150° to 250° C, the CO_2 is absorbed in $Ba(OH)_2$ and is estimated by back-titrating the excess of $Ba(OH)_2$. For the estimation of O, it is important to use low-ash coal. High sulphur content introduces error into the oxygen estimation. The bearing of the ash content on the elementary analysis, and of the reactions that are undergone in and through the ash are discussed. H. L. WHITEHEAD

1233. Determination of bitumens, humic acids, ligninic and cellulosic substances in lignites and brown coals. D. J. K. Kreulen (*Combustibles*, 1954, **14** [73], 120–123).—An air-dry 15-g sample, passed through a 0·3-mm sieve, is boiled with 300 ml of water acidified to pH 3, re-adjusted to pH 3 at intervals of 5 min., boiled until the pH remains constant, filtered off and washed acid-free. The "purified coal" (10 g) is dried at 105° C for 35 min. and extracted during 7 hr. with benzene - ethanol (96 per cent.) (1:1). The extract is evaporated and dried for 1 hr. at 105° C to give the bitumen content. The residue is dried for 15 min. at 105° C and exposed to the atmosphere for 2 hr. before weighing. To 1 g are added 300 ml of water and 20 ml of 5 per cent. aq. NaOH; the mixture is heated to boiling in 4·5 to 5 min. and boiling is continued to a total heating time of 8 min. It is cooled rapidly and transferred to a calibrated centrifuge tube, made up to 100 ml and spun for 30 min. at 2000 r.p.m. The

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solution is decanted into a 500-ml calibrated flask, and the residue washed 3 times by making up to 100 ml with water, shaking, centrifuging and decanting. The combined solution and washings are made up to volume, mixed, and a sample containing 0·5 to 1 mg is withdrawn, made up to 50 ml with water and treated with 20 ml of 33 per cent. H_2SO_4 and 20 ml of 0·01 N $KMnO_4$. It is heated to boiling in 3·5 to 4 min. and boiled for a total heating time of 10 min., cooled rapidly to 70° C and titrated with 0·01 N $KMnO_4$, after adding 20 ml of 0·01 N oxalic acid. One ml of $KMnO_4 \equiv 0\cdot10$ mg of humic acid. The residue in the centrifuge tube is shaken with 50 ml of water acidified to pH 3, centrifuged for 10 min. at 2000 r.p.m., decanted and dried; this is repeated with 50 ml of 96 per cent. ethanol and 50 ml of ether; final drying is at $< 60°$C. It is then placed in a bath at 12° to 15° C and 15 ml of 72 per cent. H_2SO_4 are added with stirring for 1 min., the temp. is allowed to rise, and the mixture is left to stand for 2 hr. with occasional agitation. The mixture is added to 400 ml of water, made up to 575 ml, refluxed for 4 hr., cooled to 50° C, filtered off through a tared porcelain filter, washed until sulphate-free and dried to constant weight. Incineration of this residue gives the ash. Cellulosic substance is determined by difference.

D. LEIGHTON

1234. Application of fluorescent X-ray spectrometry to analysis of hazardous industrial dusts. H. C. Terford (*Dissert. Abstr.*, 1954, **14** [10], 1524).—The application of fluorescent X-ray spectrometry to the determination of Fe in industrial dusts is described; this is of value in the radiographic diagnosis of siderosis. A calibration curve is established by plotting the ratios of intensity of the $Fe_{K\alpha}$ line to that of the $Ni_{K\alpha}$ line as a function of the composition, determined from homogeneous samples containing 20 per cent. of Ni as an internal standard and known quantities of Fe. Dust samples taken from all foundry operations are tested, sufficient powdered Ni being added to make the final composition 20 per cent. These tests show a high iron content for low quartz percentage, and *vice versa*. Accurate and reproducible results are obtained by this method. S.C.I. ABSTR.

See also Abstracts 1111, 1113, 1114, 1115, 1117, 1119, 1237, 1238, 1379, 1380, 1381, 1382, 1383, 1384, 1418.

3.—ORGANIC ANALYSIS

1235. Cryometric analysis of small samples of organic substances. M.-P. Mathieu (*Bull. Soc. Chim. Belg.*, 1954, **63** [8–10], 333–356).—The calculation is described of the amount of impurity in a substance from the extent of the plateau in its fusion curve. A suitable heating block and thermocouple arrangement is described and the method is proposed as suitable for routine determinations on organic substances. About 0·5 to 1 g of material is required. A. JOBLING

1236. Acetic anhydride in non-aqueous titrimetry; determination of sulphuric acid catalyst in ethanol esterification mixtures. A. F. Gremillion (*Anal. Chem.*, 1955, **27** [1], 133–136).—The procedure consists in a potentiometric titration of the H_2SO_4 in a 100-ml sample of the esterification mixture with a 0·1 N solution of Na acetate in acetic acid, after the addition of 100 ml of acetic anhydride. With mixtures containing a high amount of H_2O or

ethanol, the operation is effected in a flask fitted with reflux condenser. Total removal of H_2O and ethanol is not essential, although both these compounds in quantity influence adversely the results. Acetic anhydride can also be used as a solvent in the titration of pyridine, primary and secondary amines (titrating at 0° C), and some bases (glycine, urea, etc.) whose ionisation constants in H_2O are $\ll 10^{-15}$. The end-points are very sharp and are often characterised by large changes in potential. Typical titration curves are shown. W. J. BAKER

1237. The semi-micro determination of fluorine, chlorine and nitrogen in organic compounds. F. Brown and W. K. R. Musgrave (*Anal. Chim. Acta*, 1955, **12** [1], 29–33).—A sample containing about 10 mg of each element to be determined is fused with Na in a nickel bomb (contact with copper must be avoided) at 600° to 700° C. The excess of Na is destroyed with ethanol and the material is washed into a beaker, heated to 50° C for 2 min., cooled, filtered and diluted to 150 ml. Fluoride is determined in an aliquot of this soln., after removal of CN′, by the method of Musgrave *et al.* (*Brit. Abstr. C*, 1950, 127) or that of Belcher *et al.* (*Anal. Abstr.*, 1954, **1**, 492). Chloride is determined in a 50-ml aliquot by the following procedure. Add 0·1 M aq. formaldehyde (10 ml), neutralise to phenolphthalein with 2 N HNO_3, add conc. HNO_3 (2 ml) and 0·01 N $AgNO_3$ in excess. Filter, and titrate the filtrate and washings with 0·01 N KBr. Use 6 drops of 0·05 per cent. aq. Rhodamine BS as indicator. Nitrogen is determined by titration of the CN′ with $AgNO_3$ in a 25-ml aliquot mixed with 6 N aq. NH_3 (7 ml) and 0·1 N KI (2 drops). The method is not applicable if the substance contains oxygen, because some of the nitrogen is then converted to cyanate. W. C. JOHNSON

1238. Micro-determination of sulphur in organic compounds. M. Večeřa (*Chem. Listy*, 1954, **48** [4], 613–616).—The method of Zinneke (*Brit. Abstr. C*, 1951, 289) has been improved to give highly accurate results with 1 to 3-mg quantities of a wide range of organic sulphur compounds. The sulphur oxides formed by the combustion of the substance on a platinum contact in a current of oxygen are absorbed in a tube (10 cm long) filled with silver shavings and kept at 450° to 480° C. The Ag_2SO_4 is then extracted from the absorption tube with ten 5-ml portions of hot water and the Ag is determined by potentiometric titration with 0·01 N KI. The method has been tested on compounds containing from 6 to 43 per cent. of sulphur. G. GLASER

1239. Electrical methods (more especially polarographic) of determining carbonyl groups. Ch. Prévost and P. Souchay (*Chim. Anal.*, 1955, **37** [1], 3–15).—Methods of determining aldehydes and ketones by electrometric titration and by polarography are reviewed. Various direct methods (with $NaHSO_3$ and $LiAlH_4$) and indirect methods based on the formation of oximes and bisulphite compounds are described; the polarographic method is preferred. The mechanism of carbonyl group reduction is considered for various types of compound. The behaviour of different types of carbonyl-containing mol. shows that polarographic reduction is always a simple transfer of electrons from the electrode to the mol. undergoing reduction, and that explanations involving hydrogen atoms are not really tenable. The relationship between the half-wave potential and the constitution of the mol. is studied. A. J. MEE

1240. **The determination of hydroperoxides.** J. P. Wibaut, H. B. van Leeuwen and B. van der Wal (*Rec. Trav. Chim. Pays-Bas*, 1954, **73** [11], 1033–1036).—The iodimetric method of Robertson and Waters (*J. Chem. Soc.*, 1948, 1584) for the determination of hydroperoxides is modified by using a lower temp. to prevent interference of α-tetralone and a higher concn. of KI to ensure complete reaction. Up to 0·4 millimole of the hydroperoxide is introduced in a 1-ml conical flask into a 300-ml flask fitted with a ground-glass joint and a bubbler tube, into which 20 ml of acetic acid, 1·5 g of $NaHCO_3$ and 5 ml of 40 per cent. aq. KI are also introduced without mixing with the sample until evolution of CO_2 has been completed through the bubble trap. The contents are then mixed and the mixture is kept in the dark for 10 min. at room (or the appropriate higher) temp., diluted with 50 ml of water and titrated with 0·02 N $Na_2S_2O_3$. The method was tested with 1-tetralyl hydroperoxide (whose preparation is described), benzoyl peroxide, 1:1-diphenylethyl hydroperoxide, hydrogen peroxide, $\alpha\alpha$-dimethylbenzyl hydroperoxide and *tert.*-butyl hydroperoxide at room temp. and gives nearly quantitative results. Determinations with 9-*trans*-decalyl hydroperoxide and 2:5-dihydroperoxy-2:5-dimethylhexane give quantitative results when the reaction mixture is kept at 60° C for 10 min.   E. J. H. BIRCH

1241. **Detection of polyethylene glycols.** F. Neuwald and K. Adams (*Dtsch. ApothZtg.*, 1954, **94** [51], 1258).—The glycols of various degrees of condensation and their esters and ethers are identified by conversion by means of H_2SO_4 to dioxan, which is detected by the formation of a characteristic ppt. with aq. $HgCl_2$. For the test, 2 ml of the sample are carefully heated with a few drops of conc. H_2SO_4 and the distillate is collected in a tube half-filled with 1 per cent. aq. $HgCl_2$ solution. The ppt. formed with dioxan appears in a short time. Care is required with the test because of the inflammability and poisonous nature of dioxan.   H. L. WHITEHEAD

1242. **Detection and determination of acraldehyde in alcoholic liquids.** L. Rosenthaler and G. Vegezzi (*Z. LebensmittUntersuch.*, 1954, **99** [5], 352–361).—Colour reactions are described for acraldehyde, in the presence of trichloroacetic acid and $HgCl_2$, with resorcinol and resorcinol analogues. Six tables of colour tests relative to the acraldehyde content are given in detail. In the quantitative determination of the acraldehyde, 10 ml of liquid are used. The alcohol content is estimated first and standardised when necessary to 40 or 80 per cent. v/v. In this liquid the total aldehyde is determined with *m*-phenylenediamine (1 ml) and the acraldehyde with hexylresorcinol. The colour is compared with the corresponding standard colour solution in 40 per cent. alcohol after two hours, or in 80 per cent. alcohol after one hour. From the result of the phenylenediamine test, the figure for acraldehyde is read from the table and is subtracted; a value for acetaldehyde can be calculated.   S.C.I. ABSTR.

1243. **Analytical procedures using a combined combustion-diffusion vessel. Improved method for the degradation of ¹⁴C-labelled lactate and acetate.** J. Katz, S. Abraham and I. L. Chaikoff (*Anal. Chem.*, 1955, **20** [1], 155–156).—Simple combustion-diffusion flasks (50 ml) of the type described previously (*Anal. Abstr.*, 1955, **2**, 516) can be used for the simultaneous complete degradation of ¹⁴C-labelled lactate and acetate or other short-chain fatty acids. The lactate is oxidised with 5 per cent. $KMnO_4$ in 2 N H_2SO_4, the acetic acid being removed by steam-distillation and then titrated with NaOH, whilst the CO_2 is absorbed in $Ba(OH)_2$ in the centre well of the flask. The Na acetate solution is evaporated to dryness and the residue is dissolved in 1 or 2 ml of H_2O, aliquots being taken for (*i*) $K_2S_2O_8$ oxidation, which yields CO_2 from the C_1 and C_2 atoms of the lactate, and (*ii*) decarboxylation with NaN_3 to form methylamine, which is either burnt directly (*Anal. Abstr.*, 1955, **2**, 516) or distilled into an excess of H_2SO_4, the solution being treated with $KMnO_4$ at 90° and the CO_2 absorbed as above. Recoveries of CO_2 and methylamine are $\simeq$ 90 per cent.   W. J. BAKER

1244. **The determination of pentaerythritol as the dibenzylidene-acetal and its chromatographic separation from commercial material.** K. Sporek and A. F. Williams (*Analyst*, 1955, **80**, 29–36).—The reaction of benzaldehyde with pentaerythritol to form the dibenzylidene-acetal, which forms the basis of a method for the determination of pentaerythritol, is applicable only to fairly pure material. The likely impurities in plant liquors or unknown commercial products are formaldehyde (causing low analytical results) and liquid hydroxy compounds and dipentaerythritol (causing high results). A chromatographic procedure, with Celite as adsorbent, for separation of sufficiently pure pentaerythritol from impure samples is developed. The prepared sample is transferred to the column with dry acetone and the column is washed with this solvent. The impurities pass through and the pentaerythritol is subsequently eluted with aq. acetone. The application of the benzaldehyde procedure to this eluate is described.   A. O. JONES

1245. **New volumetric methods for the determination of organic compounds. II. Reduction method for chloroform and carbon tetrachloride by the use of chromous ion.** T. Kiba and K. Terada (*J. Chem. Soc. Japan*, 1954, **75** [2], 196–198).—$CHCl_3$ and CCl_4 can react with $Cr^{..}$ in aq. soln. made 1 to 3 N with HCl to give CH_4 and the chromic ion. The reduction of $CHCl_3$ proceeds quant. within 20 min. at 80° C and that of CCl_4 within 20 min. at 20° C. At a higher temp. the reduction of CCl_4 is much slower. $CrCl_2$ soln. can be prepared as follows. A 20-ml portion of aq. $K_2Cr_2O_7$ (29·4258 g per litre) is introduced into an amalgam reductor and reduced to $CrCl_2$ with 3 per cent. zinc amalgam in 1·5 N HCl. After the removal of Zn - Hg, 1 ml of ethanol is placed in a small cup which is attached to the top of the reductor. The sample ($\simeq$ 0·1 g) is added to the cup from a Lunge - Ray pipette and washed into the reductor with a further 1 ml of ethanol. After the reduction, the excess of Cr^{II} is titrated with standard Fe^{III} soln. (iron alum), by using KCNS as indicator (*b*). A blank test should be carried out (*a*). The amount of CCl_4 or $CHCl_3$ can be found from (*a* − *b*). The presence of alcohols, ethers and acetone does not affect the result. The weighing of the Lunge - Ray pipette before and after the dropping of the sample should be done carefully and rapidly. The mixture of $CHCl_3$ and CCl_4 can be titrated at room temp. to give the amount of CCl_4 without being affected by the presence of $CHCl_3$, provided the amount of $CHCl_3$ is not more than that of CCl_4. A similar process can be applied for the determination of oximes (*J. Chem. Soc. Japan*, 1953, **74**, 808).   K. SAITO

1246. Quantitative determination of mixtures of chloroacetic acids. J. Barceló and M. Pilar Jorge (*Inf. Quím. Anal.*, 1954, **8** [6], 198–207).—Chemical, colorimetric, chromatographic, polarographic and Raman spectrographic methods of determining chloroacetic acids are reviewed, and their limitations are indicated. L. A. O'NEILL

1247. Colour reactions of substitution products of urea. F. von Gizycki and L. Reppel (*Z. anal. Chem.*, 1955, **144** [2], 109–111).—Colour reactions of phenyl-substituted urea derivatives with H_2SO_4 containing KNO_3 are recorded. Ring-colours on initial mixing are compared with those obtained after shaking the solutions. D. R. GLASSON

1248. Quantitative determination of hexamethyl-enediamine [1 : 6-diaminohexane]. R. Carpignano and G. Musso (*Ann. Chim., Roma*, 1954, **44** [10], 821–826).—*Gravimetric method*—0·1 to 0·2 g of technical hexamethylenediamine (**I**) is dissolved in 5 ml of ethanol and treated with 2·5 ml of conc. HCl and 20 per cent. excess of aq. H_2PtCl_6 (10 per cent.) for 15 min., then the **I** chloroplatinate ($C_6H_{18}N_2.H_2PtCl_6$) is filtered off, washed with aq. ethanol until the washings give no turbidity with formaldehyde in aq. KOH solution, and dried at 100° C. Photomicrographs of the chloroplatinate and chloroaurate crystals are given. *Colorimetric method*—Aq. solutions of $\simeq$ 0·01 to 0·1 per cent. of **I** (10 ml) are treated with 0·5 ml of acetone and 0·5 ml of 0·5 per cent. sodium nitroprusside. The transmittance is measured at 530 to 550 mμ. The reagent must be protected from light. A curve is given for 0 to 0·1 per cent. of pure **I**. T. P. McLAUGHLIN

1249. Determination of acrylonitrile [vinyl cyanide] in air. J. Haslam and G. Newlands (*Analyst*, 1955, **80**, 50–53).—A method is described for the determination of vinyl cyanide in air at concn. of 0 to 150 mg per cubic metre. The air ($\simeq$ 40 litres at 2 litres per min.) is drawn through two absorbers containing a 1 per cent. soln. of lauryl mercaptan [dodecanethiol] in *iso*propanol. Ethanolic KOH is then added to the contents of the absorbers to catalyse the reaction between the thiol and the cyanide. After 4 min., glacial acetic acid is added and then a pre-determined vol. of iodine soln. ($\simeq$ 0·03 N), equiv. to the thiol originally present. The residual I is proportional to the vinyl cyanide and is determined absorptiometrically with the aid of a previously constructed calibration graph. The reaction is fairly specific although $\alpha\beta$-unsaturated nitriles, esters or aldehydes would interfere, but these are not likely to be present in the air of factories in which vinyl cyanide is being handled. The accuracy of the method ($\simeq$ 90 per cent.) is sufficient for the purpose of the determination, viz., to ascertain the concn. of the highly toxic cyanide in the air. A. O. JONES

1250. Methylimide determination. III. Behaviour of *iso*propyl iodide with wash liquids containing sodium thiosulphate and other substances. (Note on the micro-determination of glycerol.) F. Franzen, K. Eysell and H. Hack (*Mikrochim. Acta*, 1954, [6], 708–711).—The extent of the reaction of *iso*propyl iodide with a 5 per cent. aq. $Na_2S_2O_3$ soln. used as a washing liquid in the determination of alkoxyl and alkylimide groups (*Brit. Abstr. C*, 1952, 488; 1953, 506) is very small. The amount of decomposition is about 37, 8 and 1 per cent. for methyl, ethyl and *iso*propyl iodides, respectively, and can be used in

separating the alkyl groups in the respective compounds. The decomposition caused by sodium antimonyl tartrate (10 per cent.), hydroxylamine hydrochloride (5 per cent.) with Na_2CO_3, and L-ascorbic acid (5 per cent.) is also slight. The application to the determination of glycerol is also considered. A. J. MEE

1251. Identification of organic compounds. VII. Identification of sulphides. M. Jureček, M. Večeřa and J. Gasparič (*Chem. Listy*, 1954, **48** [4], 542–548).—The *p*-bromophenacylsulphonium bromides (**I**) of eleven dialkyl sulphides have been prepared by boiling an equimolecular mixture (0·01 mole) of *p*-bromophenacyl bromide (**II**) and the appropriate thioether in methanol (5 ml) during 10 to 30 min., diluting with ether and allowing the product to separate in an ice-chest. For identification purposes, the picrates (from aq. **I** and saturated aq. picric acid), and especially the perchlorates (from aq. **I** and 10 per cent. aq. $NaClO_4$), are more suitable than **I**. Boiling points of the sulphides and melting points (corrected) of **I** (recryst. from ethanol or ethanol - ether), of picrates (recryst. from conc. or aq. ethanol) and of perchlorates (recryst. from ethanol) are, respectively, in °C: dimethyl sulphide, 38°, 127°, 186°, 235°; ethyl methyl sulphide, 64° to 66°, 119° to 120°, 157°, 224·5°; diethyl sulphide, 92°, 114°, 147°, 172°; methyl *n*-propyl sulphide, 95°, 109·5°, 153°, 167° to 168°; ethyl *n*-propyl sulphide, 117°, 91° to 91·5°, 126·5° to 127°, 87°; di-*n*-propyl sulphide, 140° to 144°, 105°, 119°, 105°; *n*-butyl methyl sulphide, 122° to 123°, 101° to 102°, 121° to 121·5°, 135·5° to 136°; *n*-butyl ethyl sulphide, 142° to 144°, 99·5° to 100°, 103°, 96° to 96·5°; *n*-butyl *n*-propyl sulphide, 167° to 168°, 89°, 77° to 78°, 90°; di-*n*-butyl sulphide, 65° (at 8 mm pressure), 96·5°, 79°, 131°; *iso*butyl methyl sulphide, 110° to 114°, 105°, 148°, 176°. Some branched dialkyl sulphides do not react with **II**. A general procedure for the preparation of the sulphides is also given. G. GLASER

1252. Physico-chemical determination of phenols. II. Coulometric estimation of phenols. C. N. Van Zyl and K. A. Murray (*S. Afr. Ind. Chem.*, 1954, **8** [11], 243–245).—The coulometric titration of phenols with electrically generated Br and the apparatus used, are described. At pH 5, phenol and *o*-cresol can be determined in the range 0·6 to 0·02 p.p.m., with a standard deviation of 0·006 p.p.m. on a 50-ml sample. Results with resorcinol are not so accurate, but are satisfactory. For the determination of low concn. of phenols in sewage and industrial waste liquors containing ions that interfere with the bromination, it is proposed to remove these ions first by reaction with a soluble lead salt. A. M. SPRATT

1253. Chloramine-B as a volumetric reagent. Quantitative bromination of phenols and aromatic amines. A. Singh (*J. Indian Chem. Soc.*, 1954, **31** [8], 605–608).—The Koppeschaar method as modified by Day et al. (*Ind. Eng. Chem.*, 1928, **20**, 545) has been used with chloramine-B in the presence of KBr in acid medium. Direct titration and an external indicator method have both been used for quantitative measurements. The method is applied to several different compounds (anthranilic acid, sulphanilic acid, *m*-aminobenzoic acid, *o*-, *m*- and *p*-toluidines, *o*-, *m*- and *p*-nitroanilines, 8-hydroxyquinoline, *m*-cresol, *p*-chlorophenol and phenol). N. H. E. AHLERS

1254. Determination of glycerol trinitrate in the presence of pentaerythritol tetranitrate. M. Halse (*Medd. Norsk. Farm. Selsk.*, 1954, **16** [12], 166–169). —This method depends on the relative susceptibility of glycerol trinitrate (**I**) and the resistance of pentaerythritol tetranitrate to alkaline hydrolysis under the following conditions: the powdered sample, containing $\simeq$ 1 mg of **I**, is shaken several times during 2 hr. with 20 ml of 0·1 N NaOH at room temp. The mixture is made up to 50 ml and centrifuged. The NO_2' formed is measured colorimetrically in comparison with a standard soln. of $NaNO_2$ by the reaction with sulphanilic acid and 1-naphthylamine in acid soln. Results are accurate to within 2 per cent. of calculated values.

P. S. ARUP

1255. Iodimetric analysis of certain types of selenenyl compounds. O. Foss and S. R. Svendsen (*Acta Chem. Scand.*, 1954, **8** [8], 1351–1356).—Nitrobenzene selenenyl bromides, thiocyanates, alkoxides and amides and 2:4-dinitrobenzeneselenenyl may be analysed as follows. *Procedure*—About 0·2 millimole of the substance is weighed into a 250-ml flask and dissolved, with gentle heating if necessary, in 4 ml of ethyl acetate. After addition of 96 per cent. ethanol (10 ml) and glacial acetic acid (1 ml), the contents of the flask are swirled while 25 ml of 0·01 N $Na_2S_2O_3$ are added. After 15 min., water (100 ml) and 0·2 per cent. starch solution (10 ml) are added and the excess of $Na_2S_2O_3$ is back-titrated with 0·01 N iodine soln. A blank titration is carried out but the glacial acetic acid is added after dilution with water, not before.   N. E.

1256. Estimation of small quantities of tetrahydrofuran in aqueous solutions. H. Specker and K. Köhle (*Z. anal. Chem.*, 1955, **144** [4], 272–275). —The method depends on the separation of a pure H_2O - tetrahydrofuran azeotrope by distillation, and the determination of tetrahydrofuran in the distillate from the volume and refractive index. A Vigreux column is used in the distillation to avoid large volumes of distillate.   A. J. MEE

1257. Tar bases. III. Determination of pyridine bases. M. Hisamura, T. Okazaki and Y. Nakai (*Coal Tar, Japan*, 1954, **6**, 54–60).—Salt solutions formed between pyridine bases and strong acids were calculated to have the following theoretical pH values: pyridine (**I**) 3·188, α-picoline (**II**) 3·688 and 2:6-lutidine (**III**) 4·087. Solutions (0·1 N) of **I**, **II** and **III** were titrated with 0·1 N HClO₄, the indicators used being dimethyl yellow plus methylene blue (pH 3·28) for **I** and methyl orange plus indigo carmine (pH 4·10) for **III**. For **II** no adequate indicators were found, so the mean of two titrations with both the above indicators was taken. The pH values of perchlorates of **I**, **II** and **III** were determined with the quinhydrone electrode and were found to agree with those calculated. The method was applicable to binary systems between **I**, **II** and **III**, the errors being $\pm$ 1 per cent.

CHEM. ABSTR.

1258. Spectrochemical analysis. II. Direct reading and spectrographic analysis of lubricating oils using the rotating disc electrode. J. M. Gillette (*Dissert. Abstr.*, 1954, **14** [10], 1518).—The oil standard or sample is placed in an aluminium boat under a rotating graphite disc so that the disc dips into the oil. The rotation of the disc carries the oil into the gap, where it is excited by a unidirectional discharge of an arc-like character. A cadmium internal standard is used. Spectra are recorded on Kodak spectrum analysis No. 2 films by use of a 1·5-metre spectrograph. The two-step filter method is used to calibrate the emulsion for each new roll of film. Standard photometric methods are used to construct the working curves. No internal standard is required for Quantometer recording as the reflected beam is used for internal-standard control. Quantometer chart readings are plotted against concentrations on linear co-ordinate paper to construct working curves. Accuracy is in the order of 10 per cent. for the 12 elements determined.

S.C.I. ABSTR.

1259. Chromatography and paper electrophoresis of sulphonephthalein dyes. G. T. Franglen (*Nature*, 1955, **175**, 134).—Sulphonephthalein dyes can be readily distinguished from each other by paper chromatography and filter-paper electrophoresis, the former being more effective in most cases, but the latter enables bromophenol red to be separated from cresol red. Experiments have shown that the majority of samples tested have appreciable quantities of impurities, a fact which may be important in their use for semiquant. determination of pH. Dyes were 2 per cent. w/v soln. in absolute ethanol and 20-μl spots were applied to Whatman No. 3 MM filter-paper. The paper was equilibrated overnight with 2-methylbutan-2-ol (**I**) and NH_3 (sp. gr. 0·880) and developed by descending chromatography with **I** (200 ml) and aq. NH_3 (sp. gr. 0·880) (50 ml) for 26 hr. Alternatively, the paper was equilibrated with **I**, glacial acetic acid and H_2O, and developed by **I** - glacial acetic acid - water (200 : 50 : 50, by vol.). R_F values of the main constituent of the dyes determined by the two methods are given in tabular form together with the number of other compounds present in each dye examined. Electrophoresis was carried out in an apparatus similar to that described by the author (*J. Clin. Path.*, 1953, **6**, 183) using 0·06 M diethylbarbiturate buffer, pH 8·6, and applying a potential difference of 8·0 V per cm for 3 hr.   P. HAAS

1260. Qualitative and quantitative analysis of vat dyes by paper chromatography. E. Klingsberg (*J. Soc. Dyers Col.*, 1954, **70** [12], 563–567).—Chromatographic separations of vat dyes in leuco form on strips of paper or cellulose have been effected in nitrogen atmospheres. This technique has been applied to the quant. analysis of model mixtures. The individual constituents have been determined spectrophotometrically in dimethylformamide after their chromatographic isolation from the mixture.

E. S. LANE

1261. [Colorimetric] analysis of cystine in wool. H. Zahn and K. Traumann (*Melliand Textilbericht.*, 1954, **35**, 1069–1073).—A detailed description of the method, apparatus used and preparation of standard soln. is given. Wool samples weighing 220 to 230 mg are dried to constant wt. at 105° C and are hydrolysed with 4 ml of 30 per cent. H_2SO_4 for some hr. To 1 ml of the filtered hydrolysate in a 25-ml calibrated flask, 1 ml of standard cystine soln. and 6 ml of an acetate buffer are added and the mixture is placed in a water bath at 10° C. One ml of Na_2SO_3 soln. is added, and, after 10 min., 2 ml of Folin's reagent; the mixture is left to stand at 20° C for 30 min., then is made up to volume with distilled water and the blue colour is determined colorimetrically. Details of calculation are given. A simultaneous blank determination is also made. Descriptions are given of the application of the method to the determination of chemical

changes undergone by wool on scouring, slaughter-
ing, liming, chlorinating and in laboratory tests,
and also to the estimation of wool contents in blends,
e.g., with Perlon, and casein.   S.C.I. ABSTR.

**1262. Analysis of mixtures of silk and cellulosic
fibres.** C. Earland and D. J. Raven (*J. Text. Inst.*,
1954, **45** [12], T943–T944).—A reagent prepared by
dissolving 10 g of fused $CaCl_2$ in 100 ml of 90
per cent. w/w formic acid dissolves silk, whilst
leaving cellulosic fibres unaffected. One g of a
mixture of silk and cellulosic fibre is treated at
room temp. for 20 min. with 50 ml of the reagent,
with occasional stirring. The residual cellulose is
collected on a sintered-glass filter, washed and dried
at 110° C. With cotton, no correction factor is
necessary, but results for viscose are always
0·7 per cent. too high.   L. VALENTINE

1263. Testing of wood pulp for moisture. D. E.
Davis and A. I. M. Rodger (*Proc. Aust. Pulp. Ind.
Tech. Ass.*, 1953, **7**, 213–236).—A comparison of the
U.S. boring method and the British and Scandi-
navian wedge method is followed by details of an
investigation into the horizontal and vertical mois-
ture gradients in bales of Canadian wood pulp,
which showed that the gradients were sufficiently
great to render samples taken by the boring method
unrepresentative of the bales examined. In the
shipments examined, the boring method gave lower
moisture contents than the wedge method. A bulk
method of cutting wedges from collected sheets
by guillotining was found to save time, without
loss of accuracy, compared with hand cutting of
wedges, and to be only a little longer than the
boring method. An oven is described in which
the pulp can be weighed in the drying chamber
whilst hot, the difference in pulp temperature at
the top and bottom being $\not> 1.5°$ C.
          S. V. SERGEANT

**1264. Comprehensive analysis of sodium alkylaryl-
sulphonate detergents.** F. T. Weiss, A. E. O'Donnell,
R. J. Shreve and E. D. Peters (*Anal. Chem.*, 1955, **27**
[2], 198–205).—A modification of ASTM/D855/46T
suitable for the analysis of sodium alkylaryl-
sulphonate detergents is described. Reliable values
were obtained for sodium sulphonate, inorganic
salts, neutral oil, sodium carboxylate, free acid,
alkali and water and the average combining wt. of
the sodium sulphonate. Analysis is possible in the
presence of organic and inorganic builders, and
with minor modifications application to the analysis
of sodium alkyl sulphate detergents is possible.
          G. P. COOK

**1265. Determination of chloride in alcohol-
soluble matter in detergents.** J. Pomeranz (*Chemist
Analyst*, 1954, **43** [4], 89–90).—A 14-mm × 250-mm
column of a strongly basic anion-exchange resin
is conditioned by the alternate passage of N hydro-
chloric acid and aq. NaOH soln., and regenerated
with 450 ml of aq. NaOH soln. (containing $<$ 10
p.p.m. of Fe). After washing until the effluent is
neutral to phenolphthalein, the sample (diluted to
contain $\simeq 0.01$ M chloride) is passed through the
column at 2 ml per min.; the column is washed with
three 5-ml portions of H_2O, and then eluted with
400 ml of N NaOH at 2 ml per min., followed by a
further 100 ml at 3 to 4 ml per min. The combined
eluates are acidified with dil. nitric acid, and 5 ml
of 5 per cent. ferric alum solution and exactly
1 ml of 0·1 N potassium thiocyanate are added.
The solution is titrated with 0·1 N silver nitrate

until the red colour disappears, and 1 to 2 ml are
added in excess; the excess is then titrated with the
potassium thiocyanate solution. The method is
simple and of high precision, but rather time-
consuming.   H. P. PAGET

1266. Analysis of water-soluble synthetic soaps.
N. W. Tschoegl (*Rev. Pure Appl. Chem.*, 1954, **4**
[3], 171–206).—The wide variety of chemical and
physical forms in which soaps are obtained makes
their analysis difficult. The review covers quant.
analytical methods for anionic and cationic soaps
and detergents, qual. tests for water-soluble soaps,
and schemes for systematic qual. analysis.
          A. M. SPRATT

**1267. Some applications of infra-red spectroscopy
in the qualitative analysis of plastics.** G. Torazza
Zerbi (*Chim. e Ind.*, 1954, **36** [12], 889–893).—The
application of infra-red spectroscopic analysis in the
study and control of the composition of various
plastics is described. The infra-red spectra of ten
plastics (chiefly vinyl type) and of seven plasticisers
(dibutyl, diethyl and dipentyl phthalates; triphenyl
and tritolyl phosphates; dioctyl and dibenzyl
sebacates) are illustrated. The problems of the
recognition of polyvinyl acetate in low percentages,
of distinguishing between mixtures and co-polymers
of polyvinyl acetate and polyvinyl chloride, and
of the rapid distinction of urea- and melamine-type
resins are specially considered.   C. A. FINCH

**1268. Determination of rye flour in urea-form-
aldehyde syrups.** J. Haslam and A. R. Jeffs
(*J. Appl. Chem.*, 1954, **4** [12], 653–658).—Kings-
bury's test for the colorimetric determination of
sugar in urine, which involves measuring the colour
produced by treating the sugar soln. with flavianic
acid (2:4-dinitro-1-naphthol-7-sulphonic acid) (**I**),
has been developed to determine the content of
rye flour used as a thickener in urea-formaldehyde
adhesive syrups. A calibration curve that relates
colour readings on a Spekker absorptiometer to the
flour content of syrup is obtained. Known amounts
of flour are added to non-thickened syrup to cover
the range 0 to 8 per cent., the preparations are
hydrolysed with HCl, aliquots are treated with **I**
containing formaldehyde and the absorptiometer
reading is taken with Ilford green No. 604 filters in a
1-cm cell. The linear calibration curve that relates
indicator drum readings to flour content is used to
determine the unknown flour content in urea-
formaldehyde syrups. The method is applicable
to wheat, maize, rice and potato starches. A
method for differentiating potato starch from other
starches involves the treatment of 1 to 2 drops of
the urea-formaldehyde syrup with 0·5 ml of a
1 per cent. solution of neutral red followed by
standing, centrifuging, pouring off the liquor,
washing the starch extract with 95 per cent.
ethanol, drying and examining microscopically;
potato-starch granules, though swollen and dis-
torted, are stained brown.   G. HELMS

**1269. Quantitative analysis of urea-formalde-
hyde condensation products.** L. M. Mobers (*Plastica*,
1954, **7** [12], 598–601).—Useful estimates of the
water content of the products (powdered solids,
solutions or suspensions) can be obtained by dis-
tillation with an excess of *iso*butanol, and deter-
mination of the sp. gr. of the distillate (*cf.* Feith,
Kunststoffe, 1944, **34**, 71 and 127). Results obtained
for free-formaldehyde content in the above dis-
tillate, by oxidation to formic acid with alkaline

H_2O_2, are in substantial agreement with results from direct determinations on the samples, either by the bisulphite and potentiometric titration method of Siggia and Maxcy, carried out at 0° C (*Brit. Abstr. C*, 1948, 130), or by the hydroxylamine hydrochloride method (*cf.* Smythe, *J. Phys. Colloid Chem.*, 1947, **51**, 396); in the latter process, carried out at room temp., the HCl liberated owing to oxime formation must be progressively titrated to keep the reaction mixture as nearly neutral as possible.

P. S. ARUP

1270. Qualitative and quantitative analysis in the domain of the pigment, varnish, paint and allied industries. H. Rabaté (*Peint. - Pigm. - Vern.*, 1954, **30** [4], 291–293; [5], 383–385; [6], 471–474). —The difficulties inherent in analytical work in this field and the unavoidable limitations of the results obtained are discussed; *e.g.*, many pigments, such as Prussian blue and zinc chrome, are of indefinite composition, whilst in other cases, *e.g.*, natural $BaSO_4$ (barytes) and pptd. $BaSO_4$ (blanc fixe), the composition may be the same but the technological properties are quite different.   D. R. DUNCAN

1271. Field method for detecting mildew on paint. P. F. Klens, G. Leitner and H. D. Snyder (*Amer. Paint J.*, 1954, **38** [39], 76, 78, 80 and 82). — Attempts to identify mildew on paint by staining tests have been unsuccessful, but a bleaching test has been developed for the detection of *Pullularia pullulans*, the common black mould found on paint films. Five per cent. aq. NaOCl is applied to the spot and, after $\simeq$ 1 min., it is rinsed off with H_2O and the area is blotted without rubbing. If the area is bleached, mildew is indicated; if it remains unaffected, any discoloration is probably dirt. If the test area becomes yellow during the test (*e.g.*, with white lead paint), a solution of 10 per cent. aq. $SnCl_2$ is applied, which removes the yellow stain, and is subsequently rinsed off. Care must be taken in selection of a test area to avoid certain types of chemical staining which may give false positive tests. The test is not so effective with dark paints. Correlation with microscopic examination is > 90 per cent.   L. A. O'NEILL

1272. The comparison of inks and writings by paper chromatography. B. B. Coldwell (*Analyst*, 1955, **80**, 68–72).—Methods are described for the examination by descending paper partition chromatography of writing inks and dried ink deposits. Any chromatographic chamber capable of containing paper sheets (Whatman No. 1), 18 in. × 22 in., is suitable. A vol. (0·005 ml) of ink, preferably at several dilutions, is spotted on the starting line with the same vol. of a comparison ink 2 or 3 cm away alongside. Chromatograms are prepared with one of three solvent mixtures, *viz.*, *n*-butanol - acetic acid - water (4:1:5 by vol.), phenol - water (4:1 by wt.) or phenol - water - acetic acid (75:24:1 by wt.). These solvents require 14 to 16 hr. to descend 15 in. at 25° C. The paper is dried at room temp. With specimens of writing and other ink films, water serves as transfer solvent for fountain-pen and soluble stamp-pad inks and glycerol - acetone - acetic acid - water (2·6:3·0:2·5:10 by vol.) for ball-pen inks and pad inks insoluble in water. Ink dyestuffs can be qual. identified by comparing the ink chromatograms with those of known dyes. A more exact and positive identification of the dyes extracted from the chromatogram is described, and relative concn. can also be determined by this method. Different brands

of inks may contain the same dyes and give identical chromatograms, but different chromatograms provide strong evidence that the inks are dissimilar.

A. O. JONES

See also Abstracts 1140, 1385.

4.—BIOCHEMISTRY
INCLUDING DRUGS, FOOD, SANITATION, AGRICULTURE

Blood, Bile, Urine, etc.

1273. Estimation of sodium in serum and other biological fluids. A. Venkataraman (*J. Sci. Ind. Res., B, India*, 1954, **13** [10], 713–718).—A new reagent containing uranyl nitrate and magnesium acetate in 80 per cent. ethanol is recommended for the estimation of Na in biological fluids by the procedure outlined by Trinder (*Brit. Abstr. C*, 1952, 18). The method requires few manipulations, the isolation and washing of the precipitated triple salt being avoided. The Na and the serum proteins in 0·1 ml of the serum are precipitated simultaneously, the precipitation being complete and quantitative, even at room temp., within less than 15 min. The Na content is found indirectly by measuring the concentration of the U before and after the precipitation of the Na as the triple salt. The presence of K, even up to 200 mg per cent., does not interfere with the precipitation of the Na. Ca and Fe in concentrations of 10 mg per cent. have no effect. Small amounts of phosphates, even up to 100 mg per cent., do not affect the accuracy. The procedure is applicable to most body fluids except urine.

G. C. JONES

1274. The analysis of blood iron. R. C. Dickenman, B. Crafts and B. Zak (*Arch. Biochem. Biophys.*, 1954, **53** [2], 381–386).—In the simple method described, the blood sample is oxidised and proteins are removed by treatment with a $NaClO_3$ - $HClO_4$ reagent. After filtration into a buffered solution containing ascorbic acid and 2:2′-dipyridyl or 1:10-phenanthroline, the resulting colour is measured spectrophotometrically. *Reagents*—(i) Dissolve 300 g of sodium acetate in 700 ml of iron-free distilled water. Mix with 1 g of 2:2′-dipyridyl or 1:10-phenanthroline dissolved in 200 ml of water and dilute to 1 litre. (ii) Dissolve 1 g of ascorbic acid in 100 ml of water. Prepare freshly. (iii) Dissolve 3·2 g of $NaClO_3$ in 500 ml of distilled water. Cool in an ice-bath and pour in slowly 85 ml of 72 per cent. $HClO_4$ previously cooled in an ice-bath. (iv) Dilute 82 ml of 72 per cent. $HClO_4$ to 1 litre with water. *Procedure*—To 0·5 ml or 1·0 ml of oxalated blood in a 25-ml test-tube, add 10·0 ml of (iii). Cover the tube and place it in a bath of boiling water for 8 min. Cool in an ice-bath, wash down the sides of the tube with about 1 ml of (iv) and filter into a 100-ml calibrated flask containing 20·0 ml of (i) and 5·0 ml of (ii). Wash the test-tube and filter several times with small amounts of (iv) and dilute the filtrate to vol. with water. Measure the resulting colour at 508 mμ. Calibration, which is rectilinear with up to 600 μg of Fe, is carried out with 1·0 to 5·0-ml quantities of a standard 0·01 per cent. iron soln. by omitting the heating and filtration procedure, and with a reagent blank.

W. H. C. SHAW

1275. Vitamin-B_{12} assay in the blood of patients and normal subjects. J. M. Alés and F. Vivanco (*Bull. Inst. Med. Res., Univ. Madrid*, 1954, **7** [1], 33–39).—A turbidimetric modification of the *L.*

leichmannii (ATCC **4797**) microbiological method for vitamin B$_{12}$ is described in which 5 ml of serum are used for assay. Determinations on twenty normal subjects gave values from 50 to 560 $\mu\mu$g per ml with a mean of 226 $\mu\mu$g per ml. Levels below 100 $\mu\mu$g per ml are considered abnormal, whilst under intensive treatment with vitamin B$_{12}$ the level may reach 1500 $\mu\mu$g per ml. The sensitivity of the method described is 30 $\mu\mu$g per ml.

W. H. C. SHAW

1276. A simple procedure for the estimation of the alcohol content of body fluids for clinical and forensic purposes. I. E. Vidic (*Arzneimittel-Forsch.*, 1954, **4** [7], 411–418).—A method is described for the determination of ethanol by oxidation with a vanadic - sulphuric acid reagent; the resulting blue colour is measured photometrically. This method is claimed to be more accurate than the Widmark procedure. H. F. W. KIRKPATRICK

1277. The estimation of the alcohol content of biological fluids by means of vanadic acid. II. E. Vidic (*Arzneimittel-Forsch.*, 1954, **4** [8], 506–507). —Further observations on the reaction between ethanol and vanadic - sulphuric acid reagent are reported, and the best procedures for blood and urine are described. H. F. W. KIRKPATRICK

1278. The spectrophotometric estimation of ethanol in body fluids as acetaldehyde thiosemicarbazone. O. Schmidt and R. Manz (*Klin. Wochschr.*, 1955, **33** [3–4], 82–85).—Some theoretical considerations of the oxidation of ethanol to acetaldehyde and its estimation as thiosemicarbazone are presented, together with the design of a suitable diffusion chamber. The following technique for the estimation of ethanol in blood was evolved. Serum or whole blood (0·5 ml) is freed from protein by centrifuging after treatment with 10 per cent. perchloric acid (1 ml). Into 3 or 4 of the compartments of the outer chamber of the diffusion apparatus is placed the H$_2$SO$_4$ - K$_2$Cr$_2$O$_7$ soln. (5 ml); this soln. contains K$_2$Cr$_2$O$_7$ (5 g), chromic chloride (0·5 g) and N H$_2$SO$_4$ (100 ml). In one of the empty compartments is placed the test solution (0·2 ml). In the inner chamber is placed 0·001 M thiosemicarbazide soln. (5 ml) (prepared from a stock soln. of 0·1 N thiosemicarbazide) in 0·1 N H$_2$SO$_4$. After closing the chamber, the contents of the compartments are mixed and the chamber is maintained at 30° C for 3½ hr. The absorption of the thiosemicarbazone formed is estimated at 261·5 mμ in a quartz cuvette. Suitable blank controls are also run and a direct calibration curve is established with ethanol. Errors are given as follows: 75 estimations of three concentrations of alcohol gave the following values for standard deviations: 0·74 $\pm$ 0·016 g per litre; 1·47 $\pm$ 0·021 g per litre; 2·95 $\pm$ 0·047 g per litre. G. W. CAMBRIDGE

1279. A modified method for the enzymatic micro-determination of ethanol. N. G. Brink, R. Bonnichsen and H. Theorell (*Acta Pharmacol. Tox., Kbh.*, 1954, **10** [3], 223–226).—A simple rapid method (the "ADH method," see *Anal. Abstr.*, 1955, **2**, 131) is based on the reaction between ethanol and diphosphopyridine nucleotide (DPN) in the presence of alcohol dehydrogenase (ADH) to form acetaldehyde and reduced diphosphopyridine nucleotide (DPNH), which is determined spectrophotometrically. Calibration is rectilinear with 1 to 10 μg of ethanol and up to 40 μg may be estimated by the use of a standard curve. The mean error is about 4 per cent. *Reagents*—(*i*) Dissolve the equivalent of

100 mg of pure DPN in 10 ml of 0·1 M glycine - NaOH buffer at pH 6. (*ii*) Dissolve 125 mg of semicarbazide hydrochloride, dried to remove traces of ethanol, in 20 ml of 0·1 N NaOH. Adjust to pH 10 with aq. NaOH and dilute to 200 ml with 0·1 M glycine - NaOH buffer at pH 9·6. (*iii*) Aq. ADH containing about 1 mg of enzyme per ml. (*iv*) Standard aq. ethanol solutions containing 1 to 10 μg of ethanol in 0·05 ml. *Procedure*—Mix together 1·30 ml of (*i*), 35·10 ml of (*ii*) and 2·60 ml of (*iii*) and allow to stand at room temp. for 30 min. Into each of the required number of (Beckman) cells, pipette 3·00 ml of this mixture and read each cell against one (the blank) at 340 mμ. Add 0·05 ml of water to the blank and 0·05 ml of test or standard soln. to the others. Mix the contents of each cell, allow to stand for 30 min. and then again read each cell against the blank. The increase in reading at 340 mμ is proportional to the amount of ethanol, which is calculated by reference to a calibration graph (prepared each day) or with a proportionality factor. W. H. C. SHAW

1280. Experimental investigation of the behaviour of Veronal (barbitone), Luminal (phenobarbitone) and Phanodorm (cyclobarbitone) in chromatographic adsorption analysis [of urine]. W. Paulus and H. J. Mallach (*Arzneimittel-Forsch.*, 1954, **4** [6], 391–392). —Solutions of the pure barbiturates in methanol, ethanol, ether, acetone and chloroform showed no adsorption with silica-gel columns, but other materials adsorbed the barbiturates in one or more of these solvents. Purification of urine extracts by adsorption on alumina followed by elution with another solvent was not satisfactory; the best results were obtained by passing the ether or chloroform extract of urine through a silica-gel column, when no barbiturate was retained and the product was pure. Ether extracts gave a purer residue than those of chloroform. H. F. W. KIRKPATRICK

1281. A new colorimetric method for the estimation of 4-aminophenazone in serum in the presence of Pyramidon [amidopyrine]. R. Haslinger and W. Strunz (*Arzneimittel-Forsch.*, 1954, **4** [4], 299).— Dilute 2 ml of serum with 2 ml of water, add 1 ml of acetate buffer (56·6 ml of acetic acid and 118·0 g of Na acetate in water to 1 litre) and warm for 5 min. in a water bath. Filter off the pptd. protein with two 2-ml washings of hot water. To the filtrate add 2 ml of aq. NH$_3$ (3 per cent.), 0·025 ml of 5 per cent. aq. phenol soln. and 0·025 ml of 4 per cent. aq. K$_3$Fe(CN)$_6$ soln. and shake out the red colour with 3 ml of CHCl$_3$. Filter the CHCl$_3$, measure the absorption and obtain the 4-aminophenazone content by means of a standard curve. The working range is 0·2 to 10 mg per cent.

H. F. W. KIRKPATRICK

1282. The identification of *iso*nicotinic acid hydrazide in urine. F. Leuschner (*Arzneimittel-Forsch.*, 1954, **4** [11], 686).—*Iso*nicotinic acid hydrazide (**I**) can be identified and estimated in urine by two-dimensional chromatography with the use of polar solvents (*Arch. exper. Path. u. Pharmakol.*, 1954, **221**, 323) followed by contact photography in u.v. light (254 mμ). The method can detect 1 μg of **I**. H. F. W. KIRKPATRICK

1283. The lactic acid dehydrogenase in human serum. B. Hess and E. Gehm (*Klin. Wochschr.*, 1955, **33** [3–4], 91–93).—The method for the estimation of lactic acid dehydrogenase depends on oxidation of the reduced form of diphosphopyridine nucleotide (DPN) in the presence of pyruvate. The

following quantities of reagents have been found suitable for the routine determination of lactic dehydrogenase activity in serum, the reaction being started by the addition of the pyruvate and being followed in a spectrophotometer at 340 or 366 mμ: serum (1 ml), reduced DPN (0·5 μM), pyruvic acid (3 μM) and 0·05 M tri(methoxyamino)methane - HCl buffer, pH 7·5, to give a final volume of 3 ml. The initial velocity of the reaction is proportional to the enzyme concentration. Values obtained in normal subjects are compared with those obtained in subjects with pernicious anaemia.

G. W. Cambridge

1284. The diphenylamine test on leukaemic sera. A. L. Waldo and R. E. Zipf (*Experientia*, 1954, **10** [11], 468–469).—The Dische diphenylamine test is a useful aid in diagnosing leukoproliferative disorders. Only 0·2 ml of serum is used in the analysis to allow its use for samples from infants. The optical density of the clear purple reaction mixture is read from a Beckman Model B spectrophotometer at 530 mμ against distilled water as the reference. The optical density of the soln. multiplied by 1000 gives the diphenylamine (DPA) index. This index ranged from 180 to 350 units, mean 248 units, in 500 normal controls, and from 298 to 600 units, mean 448 units, in 78 leukoproliferative conditions. The range 303 to 406 DPA index units for malignant neoplasms indicates that the test might also be useful in screening for malignancy.

R. S. Tonks

1285. Assay of oxytocin in blood. G. W. Bisset and J. M. Walker (*J. Physiol.*, 1954, **126**, 588).—A method is described for obtaining concentrated extracts of blood, which can be assayed for oxytocic activity on isolated uteri of rats. *Procedure*—Arterial blood is obtained from anaesthetised heparinised rats. The blood is mixed with ethanol (9 vol., 80 per cent. v/v) and N H_2SO_4 is added until the solution is blue to Congo red. The mixture is set aside for 3 hr. at room temp. with frequent shaking. The ppt. is removed by centrifuging and the supernatant liquid is concentrated *in vacuo* to about one-tenth of its original vol., the temp. of the bath not exceeding 45° C. The extract is then centrifuged and the ppt. is discarded. The flask is washed with small amounts of the ethanol, and the washings and supernatant liquid are transferred to a clean flask and evaporated *in vacuo* almost to dryness. The residue is taken up in about 10 ml of distilled water, centrifuged and the supernatant liquid is left overnight at room temp. in an open Petri dish to evaporate to dryness. The new residue is dissolved in 5 ml of distilled water and dialysed in a cellophane membrane to remove potassium. The extract is neutralised, after dialysis, with N Na_2CO_3 soln. Experiments showed at least a 90 per cent. recovery. The extracts are assayed on the isolated uterus of the rat by Holton's method. A control specimen of every extract is treated with 0·01 M sodium thioglycollate at pH 7·5 for 30 min., specifically inactivating the pituitary hormones.

G. F. Somers

1286. An ultra-violet spectrophotometric method for the determination of 4-aminosalicylic acid in blood serum and urine. R. Gösswald (*Arch. exper. Path. u. Pharmakol.*, 1954, **222**, 220–222).—Serum (1 ml) is diluted to 6·25 ml with normal saline; the diluted soln. (1 ml) is pptd. with 96 per cent. ethanol (3 ml) and, after 5 min., centrifuged at 3000 rev. per min. for 5 min. The absorption of the colourless soln. at 265 mμ is measured in a

Beckman spectrophotometer. The concn. of 4-aminosalicylic acid is calculated by comparison with a standard curve.   N.E.

1287. Multiple chromatograms for detection of amino acids in blood serum. H. ven Horst and Y. Carstens (*J. Chem. Educ.*, 1954, **31** [11], 576).—Details are given for the construction from simple materials of a piece of apparatus whereby 2 to 16 chromatograms can be run simultaneously and under identical conditions. A modification of the circular technique of Giri *et al.* (*Lancet*, 1952, **263**, 562) was used in the separation and detection of amino acids in pathological and normal blood sera. Comparisons may be made under identical conditions with controls prepared from pure amino acids.

A. Ledwith

1288. The fractional estimation of 17-ketosteroids and corticoids in urine of women with genital carcinoma. C. Sievert and A. Puck (*Klin. Wochschr.*, 1955, **33** [3–4], 57–59).—The fractionation of ketosteroids in urine was based on the methods of Dingemanse (*J. Endocrin.*, 1946, **6**, 535) and Spiegelhoff (*Acta Endocrinol.*, 1954). A portion (500 ml) of a 24-hr. specimen of urine was extracted with benzene before and after hydrolysis and washed with Na_2CO_3 soln., NaOH soln. and water. The extract was then reduced to dryness, taken up in benzene (50 ml) and passed through a column of Al_2O_3 (16 g). Forty-six fractions were collected in the following solvents: benzene (50 ml × 7), benzene containing 1 per cent. absolute ethanol (50 ml × 24), benzene containing 0·5 per cent. absolute ethanol (50 ml × 10), benzene containing 2 per cent. absolute ethanol (50 ml × 3) and finally absolute ethanol (50 ml). The eluates were evaporated to dryness *in vacuo* and the Zimmermann reaction was carried out. The values were compared with a standard curve for dehydro*iso*androsterone. The fractions were grouped as follows: 1 to 3, artefacts produced in the extraction hydrolysis; 4 to 9, i-androstanolone and androsten-3-α-ol-17-one (only obtained under strictly neutral conditions); 10 to 15, dehydro*iso*androsterone and i-androsterone (3-β-17-ketosteroid); 16 to 20, the androsterone fraction; 21 to 32, the aetiocholanolone fraction; 33 to 35, the 11-oxyandrosterone fraction; 36 to 39, the 11-oxyaetiocholanolone fraction; 40 to 46, artefacts and unknown steroids. A comparison of these fractions obtained from the urine of normal women and from women with genital carcinoma is reported.

G. W. Cambridge

1289. A method for the estimation of factor VII-inhibitor activity in plasma and serum. J. Jürgens (*Klin. Wochschr.*, 1955, **33** [5–6], 143–144).—Factor VII-inhibitor activity may be estimated by its effect on the clotting time of a system containing Factor VII, the plasma being treated with $BaSO_4$ to remove intrinsic Factor VII. A purified Factor VII prepared according to Duckert, Koller and Matter (*Proc. Soc. Exp. Biol. Med.*, 1953, **82**, 259) is used as a standard.   G. W. Cambridge

1290. Photometric determination of arginine. P. M. Strocchi and P. Drago (*Ann. Chim., Roma*, 1954, **44** [10], 836–842).—The method is adapted from Sakaguchi's colour reaction with NaOBr. Urea is added before the NaOBr solution. For pure solutions, or fractions from protein hydrolysates, 6 ml of arginine solution (containing 0·033 to 0·1 mg) are treated with 1 ml of 10 per cent. aq. NaOH, 1 ml of 0·25 per cent. 1-naphthol in ethanol, 1 ml of 40 per cent. aq. urea and 1 ml of

NaOBr (containing 5 per cent. aq. NaOH with 2·5 per cent. aq. Br). The colour remains constant at 18° C for at least 6 hr., and is measured at 463 mμ. T. P. McLaughlin

1291. Experimental contribution to the estimation of very small amounts of protein in biological material. J. Führ and O. Süel (*Klin. Wochschr.*, 1955, **33** [3–4], 89–90).—The Führ and Hinz method (*Klin. Wochschr.*, 1953, **31**, 153) for the estimation of protein depends on the staining of the proteins with Sudan black 10B and subsequent colorimetric determination. It is claimed to have advantages over other colorimetric methods in that the Beer - Lambert law applies over a wider range of concentrations, the calibration curve being linear between 0 and 450 mg per cent. in the example given. The sensitivity of the method has been determined and it is twice (ninhydrin) to ten times (micro-Kjeldahl) more sensitive than other methods. The correction of such estimations for variations in the albumin to globulin ratio is discussed. G. W. Cambridge

1292. Absorption measurement of the coloured protein bands in paper electrophoresis with the Unicam spectrophotometer SP 500. W. F. Bon (*Chem. Weekbl.*, 1955, **51** [5], 68–69).—A simple method of measuring the absorption of the colour boundaries obtained with protein solutions and Amido black 10B or bromophenol blue in paper electrophoresis is described. A holder for the paper strip is described that can be fitted into the filter space of a Unicam u.v. spectrophotometer. With this apparatus, results with 100-mm × 10-mm paper strips and only 5 cu. mm of protein solution are good. A. J. Mee

1293. Test of the purity of proteins by ultracentrifugation. H. Dieu and J. Oth (*Bull. Soc. Chim. Belg.*, 1954, **63** [8–10], 424–435).—The flattening of the optical diagram obtained by sedimentation in the ultra-centrifuge of a protein solution is due to diffusion and polydispersion, but for a homogeneous (monodisperse) system a plot of log A/H (A = area of sedimentation diagram, H = max. height of diagram) against log t (t = time of sedimentation) has slope $\frac{1}{2}$ (diffusion only); this increases up to 1 for a heterogeneous system. The method is used for ovalbumin and bovalbumin, whose sedimentation constants and iso-electric points are close, and although only one peak is obtained in the diagram, the heterogeneity is shown. The test is also applied successfully to the components of crude papain. E. J. H. Birch

1294. Paper chromatography of various phosphates and in particular the phosphorylated derivatives of adenosine. J. Guérin (*Bull. Soc. Chim. Biol.*, 1954, **36** [10], 1453–1459).—A method of descending chromatography on paper is described for the detection and determination of (i) phosphorylated derivatives of adenosine, Na_3PO_4 and $Na_4P_2O_7$, and (ii) the three phosphates of inosine and of phosphoenolpyruvic acid. The method, which is very sensitive, will separate and detect 10^{-9} to 10^{-8} M of the compound. The solvent is a mixture of *iso*propanol and butanol in aq. NH_3; temp. and time have little effect and determinations have been carried out from 13° to 28° C, and during 17 hr. to 5 days. The compounds are detected on the chromatogram by standard methods and determination is effected by measurement of the u.v. absorption of the spots. J. N. Ashley

1295. A new modification of a method for determination of antidiuretic activity. V. Holeček, H. Polák, J. Bláha and M. Jirásek (*Endokrinologie*, 1954, **32** [1–2], 38–45).—A modification of the method of Jeffers *et al.* (*Brit. Abstr., A III*, 1942, 810) for the determination of antidiuretic activity is described. The test solution is administered intravenously, by means of a cannula, into rats rendered diuretic by administration by stomach tube of alcohol and water. The new method is very sensitive and 100 per cent. response is obtained with doses as small as 15 micro units of adiuretin A single rat can be used for 10 to 14 titrations compared with 4, at the most, using Jeffers' method. J. N. Ashley

1296. A chemical procedure for determination of ¹⁴C-distribution in labelled glucose. B. Boothroyd, S. A. Brown, J. A. Thorn and A. C. Neish (*Canad. J. Biochem. Physiol.*, 1955, **33** [1], 62–68).—A procedure based on the work of Jackson and Hudson was developed for the degradation of methyl-α-D-glucopyranoside formed from 2 millimoles of glucose. Oxidation of the glucoside with periodic acid gave C-3 (the number refers to the position of the C atom in the glucose molecule) as formic acid and a dialdehyde which was converted to the strontium salt of D′-methoxy-D-hydroxymethyldiglycollic acid. The purified salt was hydrolysed to glyoxylic and glyceric acids. The glyoxylic acid was isolated as the 2:4-dinitrophenylhydrazone (C-1 + C-2); this was decarboxylated by heat to give CO_2 from C-2. The glyceric acid was oxidised by periodate to give C-4 as CO_2, C-5 as formic acid and C-6 as formaldehyde. This degradation permitted the determination of ¹⁴C in each position of the glucose molecule, the activity in C-1 being determined by difference. The method was applied satisfactorily to glucose-1-¹⁴C and a sample of glucose labelled in all positions. C. H. Whitton

1297. A new reagent for the histochemical and chemical detection of calcium. S. M. McGee-Russell (*Nature*, 1955, **175**, 301–302).—The use of a purified dyestuff (known in impure form as "kernechtrot" or Nuclear fast red, and here called "calcium red") for the histochemical and chemical detection of Ca is outlined. Calcium red can be used to distinguish between tap-water and distilled water; it appears to show selectivity between the alkaline-earth metals and aluminium and magnesium, and might be used for gross staining of skeletal structures. C. H. Whitton

See also Abstracts 1124, 1306, 1310, 1419.

Drugs

1298. Infra-red spectroscopy and its application to pharmaceutical analysis. W. C. Price (*J. Pharm. Pharmacol.*, 1955, **7** [3], 153–166).—A review is given of fundamental principles of infra-red spectroscopy and its applications in the investigation and estimation of pharmaceutical substances, *e.g.*, sterols and derivatives, antibiotics, insecticides, vitamins, alkaloids, barbiturates, antihistamines, bacteria, etc. C. H. Whitton

1299. Studies on the assay of [the U.S.] National Formulary IX for total alkaloids of cinchona, and several suggested improvements. J. Moreno and H. M. Burlage (*Drug Standards*, 1954, **22** [7–8], 148–155).—Numerous sources of error in the assay of cinchona by the method of N.F. IX are described,

and several alternative methods are proposed. No improvements are suggested in the procedure for extracting the alkaloids from the crude bark. The use of only one reagent (Valser's) as a test for complete extraction of alkaloids is considered insufficient. Advantages of the proposed methods of assay include greater rapidity, use of fewer and smaller separating funnels, elimination of emulsions in the separations, prevention of solubilisation of alkaloids in fatty impurities and *vice versa* and separation of crystalline and amorphous alkaloids.

A. R. ROGERS

1300. A double-coloration test for the differentiation of opium alkaloids. Shu-Sing Cheng (*J. Amer. Pharm. Ass., Sci. Ed.*, 1954, **43** [12], 767–769).—A combination of Marquis's and Mandelin's reagents is suggested for the differentiation of the opium alkaloids. *Procedure*—Several minute crystals of the alkaloid are treated on a white tile with 1 drop of Marquis's reagent (2 to 3 drops of 40 per cent. formaldehyde soln. with 3 ml of conc. H_2SO_4) followed by a drop of Mandelin's reagent (1 g of finely ground ammonium vanadate in 200 g of conc. H_2SO_4), when a typical coloration occurs. Aqueous alkaloidal solutions may be first extracted before applying the test. Chlorides, carbonates, oxalates and strong reducing agents interfere.

G. R. WHALLEY

1301. A very sensitive reaction of diacetylmorphine. H. Wachsmuth (*J. Pharm. Belg.*, 1953, **8** [11–12], 569).—Diacetylmorphine ($\simeq$ 10 μg in 10 ml of H_2O) gives a violet colour with 3 per cent. aq. hydroxylamine hydrochloride (1 ml) and N NaOH (1 ml), followed by N HCl (1 ml) and 30 per cent. aq. $FeCl_3$ (2 drops). The colour is not given by morphine, codeine, papaverine, ethylmorphine (Dionin) or dihydroxycodeinone (Eucodal). By this reaction, 1 to 5 mg of diacetylmorphine can be determined quant., even in the presence of a 30-fold excess of codeine or ethylmorphine, by measuring the extinction at 500 mμ.   A. R. ROGERS

1302. Conductimetric titration of anabasine and lupinine mixtures. V. V. Udovenko and L. A. Vvedenskaya (*Tr. Sredneaziatskogo Un-ta*, 1953, No. 40, *Khim. Nauki, Kn.* 5, 3–8; *Referativnyǐ Zh., Khim.*, 1954, Abstr. No. 29,347).—Anabasine, lupinine or mixtures of both bases can be titrated conductimetrically in acetone soln. against 0·05 to 0·07 N H_2SO_4, HNO_3 or 2-naphthalenesulphonic acid, the last giving the best results. In the determination of anabasine, either of the two inflexions (corresponding to the neutralisation of the piperidine and the pyridine nitrogen bases) can be used.   E. HAYES

1303. Estimation of argemone alkaloids by the colorimetric molybdosilicic acid method. A. C. Roy (*J. Indian Chem. Soc., Ind. News Ed.*, 1954, [2], 144–146).—The toxic alkaloids sanguinarine (**I**) and dihydrosanguinarine (**II**), present in argemone oil, can be estimated quantitatively by colorimetry. *Procedure*—Various amounts of pure **I** hydrochloride were precipitated with a 5 per cent. solution of molybdosilicic acid, each precipitate was thoroughly washed, and the Mo contents were estimated colorimetrically (against standard Mo solutions) as the thiocyanate in a Spekker absorptiometer. The results showed that the ratio of Mo to **I** hydrochloride is linear and that the weight ratio in the complex is 1 to 1·66. In the application of the method to the estimation of alkaloids in argemone oil, the oil is repeatedly extracted with HCl, the

combined extracts are shaken with ether to remove fatty matter and are then treated with $FeCl_3$ to convert **II** into **I**, which is then pptd. as the molybdosilicate complex and assayed against a standard Mo solution. The average of several estimations on argemone oil was 5·7 mg of Mo, equiv. to 9·46 mg of **I** per g of oil. The method can be used for assaying as little as 0·02 g of argemone oil, pure or mixed with mustard oil, and is reasonably accurate in estimating 0·2 to 50 mg of **I** hydrochloride.

G. HELMS

1304. A procedure for estimating the racemisation of adrenaline or noradrenaline in dilute solution by means of an ion exchanger. H. Hellberg (*J. Pharm. Pharmacol.*, 1955, **7** [3], 191–197).—A procedure is described for determining the degree of racemisation of adrenaline and noradrenaline in dilute solutions. The base is adsorbed on ammonium-saturated cation-exchange resin of carboxyl type (Amberlite IRC-50) and subsequently eluted in a concentrated form by 2 M hydrochloric acid. The optical rotation and the adrenaline content of the concentrated solution are then determined by the usual methods. The interfering anions, sulphite and tartrate, are almost entirely removed from the adrenaline during the exchange process. The results of the proposed method are subject to an error of 3 to 5 per cent.

C. H. WHITTON

1305. A rapid method for the estimation of rutin content. W. Brune (*Arch. Pharm., Berlin*, 1954, **287** [8], 432–439).—Rutin in plant extracts may be estimated as follows: To 1 ml of aqueous extract are added 3 ml of a soln. containing $AlCl_3$ (16 millimoles), potassium acetate (molar) and acetic acid (16·6 millimoles) and 3 ml of lead acetate soln. (0·25 molar), giving a more or less distinct ppt. Similar treatment of a standard rutin soln. followed by stirring and titration with aq. NaOH until a permanent ppt. is formed is followed by the addition of this quantity of NaOH to both the treated extract above and a blank soln. All are then centrifuged and the extinction of the various diluted mother liquors is determined at 413 mμ after 10 and before 120 min.   F. R. MUMFORD

1306. Colorimetric reaction for estimation of cortisone, hydrocortisone, aldosterone and related steroids. Irwin Clark (*Nature*, 1955, **175**, 123–124).—It was shown by Stacey *et al.* (*Nature*, 1946, **157**, 740) that one of the substances responsible for the development of colour in the Dische reaction with diphenylamine (**I**) is ω-hydroxylaevulaldehyde (**II**). The formulae of cortisone and hydrocortisone contain reaction groups similar to those of **II**, both having terminal ketol groups and a carbonyl group 5 or 6 carbon atoms distant from the primary alcohol group. The reaction has accordingly been applied to 21 different steroids (200 μg in 3·0 ml) and the colours produced are recorded in tabular form. Reagent mixture A is glacial acetic acid (54 ml), conc. H_2SO_4 (6 ml), dist. H_2O (10 ml) and **I** (600 mg). Blank mixture B is the same as A but without **I**. For 10 to 50 μg of steroid, 1 ml of reagent is used; for 50 to 400 μg, 3 ml. Two equal aliquots of sterol soln. are evaporated to dryness in test-tubes. To one is added A and to the other an equal vol. of B; after heating in a boiling-water bath for 1 hr. they are observed in a Beckman spectrophotometer model B or DU and their optical densities are determined. The difference between the values is a measure of the amount of steroid present. Steroids with a hydroxyl group in

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position C_{21} and carbonyl groups at C_{11} and C_{20} give, in common with cortisone and similar steroids, a violet colour; those like hydrocortisone having groups at C_{21} and C_{11} or C_{17} and a carbonyl group at C_{20} give green colours. P. HAAS

1307. A method for the estimation of benzylpenicillin. G. B. Selzer and W. W. Wright (*Antibiot. & Chemother.*, 1954, **4** [12], 1196–1201).—In the given method, which was developed primarily for the assay of benzathinepenicillin (**I**), the benzylpenicillin is degraded quant. to phenylacetic acid by heating with strong aq. NaOH. After acidification, the phenylacetic acid is extracted with chloroform and determined spectrophotometrically. The procedure is also applicable to benethaminepenicillin (**II**), procaine penicillin (**III**), sodium penicillin (**IV**) and potassium penicillin (**V**), and can be used for materials low in benzylpenicillin content. The method gives results which compare well with those given by FDA methods to which it is considered superior for **I**. *Procedure for benzathinepenicillin*— (*i*) Weigh accurately about 65 mg into a 2-cm × 10-cm test-tube, add 5·0 ml of 10 N NaOH and heat in a bath of boiling water for 15 min. with occasional swirling. (*ii*) Add 1 ml of distilled water and mix by swirling. Add 5 ml of benzene, mix by swirling and allow the benzene to boil for 15 sec. Remove from the bath, centrifuge and remove the benzene by pipette. Add another 5 ml of benzene and repeat the heating, centrifuging and removal procedure. Replace the tube in the bath. (*iii*) After 90 min., restore the vol. to 5 ml with distilled water and chill in an ice-bath. (*iv*) Add 3·0 ml of 18 N H_2SO_4 dropwise with agitation and wash the contents of the tube into a separating funnel with 3·0 ml more of 18 N H_2SO_4 and 20 ml of distilled water. (*v*) Extract the soln. with three 15-ml amounts of $CHCl_3$. Filter each extract through $CHCl_3$-moistened absorbent cotton (previously washed with $CHCl_3$ and dried). Dilute the filtrate to volume in a 50-ml calibrated flask with water-washed $CHCl_3$ and measure the extinction (*A*) of this solution at 253 (max.), 255 (min.), 259 (max.), 263 (min.) and 265 (max.) mμ against a blank of $CHCl_3$ previously washed with an equal vol. of water and filtered through absorbent cotton. (*vi*) Calculate the purity from the three absorption-peak values by the following formula.

Benzylpenicillin content (per cent.) (calculated as the particular penicillin salt under test)—

$$\frac{100\,A}{\text{Grams of sample} \times 2 \times E}$$

where E = theoretical $E_{1\,cm}^{1\%}$ values at 253, 259 and 263 mμ for the particular penicillin salt (3·00, 3·72 and 2·83, respectively, for **I**). *Modifications for other salts*—For **II**, **III**, **IV** or **V** weigh about 70, 75, 45 or 50 mg, respectively. For **IV** and **V** omit step (*ii*). For **III** omit (*ii*) and in (*iv*) use a total of 16·0 ml of 18 N H_2SO_4 and limit the amount of water to 4 ml. W. H. C. SHAW

1308. Spectrophotometric determination of erythromycin in pharmaceutical products. N. R. Kuzel, J. M. Woodside, J. P. Comer and E. E. Kennedy (*Antibiot. & Chemother.*, 1954, **4** [12], 1234–1241).— The method is based on the increased absorption at 236 mμ observed after mild alkaline hydrolysis of erythromycin (**I**) base. Solutions of **I** previously exposed to mild acid conditions do not show the increased absorption when subsequently treated with alkali and such solutions are used as blanks

in the procedure described. Calibration is rectilinear between 10 and 75 μg of **I** per ml of final solution and results compare well with those for microbiological assays both on fresh and deteriorated soln. Modified procedures are given for use in the presence of "triple" sulphonamides and for the determination of **I** ethyl carbonate and **I** glucoheptonate. *Reagent*—Dissolve 42 g of reagent-grade trisodium phosphate in 125 ml of 0·5 N NaOH and 100 ml of distilled water by warming. Cool, dilute to 250 ml and filter if necessary. *Standard*— Weigh accurately about 35 mg of standard **I** (dried *in vacuo* (10 to 40 mm of mercury) at 70° C for 4 hr.) and transfer quant. to a 250-ml flask with a total of 50 ml of methanol. Dissolve the sample and dilute to 250 ml with water. *Procedure*— Pipette 10-ml portions of standard soln. in each of four 25-ml calibrated flasks. For the blank soln., add 1 ml of 0·05 N H_2SO_4 to two of the flasks, mix and set aside for 60 $\pm$ 5 min. Meanwhile, to the other two flasks, add 2 ml of the reagent and heat in a water bath at 60 $\pm$ 2° C for 15 min. Cool to room temp. in an ice-bath, dilute with water to 25 ml, mix and determine the extinction in a 1-cm cell at 236 mμ against water. To the two flasks previously set aside, add 1 ml of 0·05 N NaOH and continue with the alkaline hydrolysis and extinction measurements as above. Deduct the blank extinction value from that for the standard. Prepare a calibration graph with different amounts of the standard **I** in 10 ml of soln. and treat 10-ml volumes of test soln. containing 250 to 1875 μg of **I** in the same way as the standards. W. H. C. SHAW

1309. Colorimetric determination of viomycin. A. W. Jackson (*Antibiot. & Chemother.*, 1954, **4** [12], 1210–1215).—The diacetyl reagent of Kawerau (*Sci. Proc. Roy. Dublin Soc.*, 1946, **24**, 63) for ureido compounds is adapted for the colorimetric determination and identification of viomycin. Calibration is rectilinear with 30 to 240 μg of viomycin and the results agree well with those given by a turbidimetric biological assay. Polymyxin B, bacitracin, chloramphenicol and streptomycin do not interfere. Carbomycin, erythromycin and benzylpenicillin give slight colours, whilst chlortetracycline and oxytetracycline interfere as a result of light absorption by the intact antibiotic. *Reagents*—(*i*) Dissolve 1 g of diacetylmonoxime (butane-2:3-dione-2-oxime) in 100 ml of 5 per cent. aq. acetic acid. (*ii*) Mix 1 vol. of conc. H_2SO_4 with 3 vol. of H_3PO_4 (sp. gr. 1·75). *Procedure*—Mix thoroughly in an 18-mm test-tube, 4 ml of aq. sample soln. with 2 ml of (*i*) and 3 ml of (*ii*). Heat in a bath of boiling water for 2·5 hr., cool and determine the extinction at 475 mμ against a reagent blank. For the highest accuracy, viomycin standards should be included in each group of assays. W. H. C. SHAW

1310. The use of resistant organisms for the assay of antibiotic mixtures in preparations and body fluids. S. Friedman and A. Kirshbaum (*Antibiot. & Chemother.*, 1954, **4** [12], 1216–1221).—Methods are given for the assay of bacitracin in the presence of dihydrostreptomycin (**I**) and for the determination of penicillin in serum also containing **I**. Details are given for the preparation of strains of *Micrococcus flavus* and *Sarcina lutea* resistant to 500 μg and 100 μg per ml of **I**, respectively, for use in the two procedures. Results by the proposed methods are compared with those given by conventional cup-plate assays. W. H. C. SHAW

1311. The estimation of volatile oils in drugs. U. Hagenström (*Ätherische Öle*, 1954, **4** [11], 259–261).—The need for a uniform method and apparatus for the distillation estimation of the ethereal oils in drugs is discussed. The author uses the official apparatus of B.P. 1953 by Middleton and Cocking. Although no process of estimation gives a really complete oil yield even after a long distillation (up to 30 hr.), a distillation time of 8 hr. (or less for some oils) is considered adequate. With the apparatus of B.P. 1953, a study is made of the addition of NaCl, H_2SO_4 and NaOH to the liquors to accelerate the distillation. A comparative series of tests is made by use of water, 25 per cent. aq. NaCl (**I**), 0·1 N H_2SO_4 (**II**) and 0·1 N NaOH (**III**). **I** gives a low yield in most cases and is therefore useless. **II** and **III** sometimes give a higher yield and sometimes a lower one. Comparative tests with decalin (0·28 to 1 per cent. v/v) added to the distillation liquor (water) show that the yield is increased by the water-soluble volatile matter retained by the decalin. To avoid error the estimations should be made without any interruption in operation.   H. L. WHITEHEAD

1312. Photometric estimation of *p*-aminobenzoic acid in medical preparations of Novocain [procaine hydrochloride]. J. Richter (*Arzneimittel-Forsch.*, 1954, **4** [11], 686–687).—Mix 2 ml of test soln. with 5 ml of N NaOH in a small separating funnel and shake vigorously for 30 sec. with 20 ml and then 10 ml of $CHCl_3$; discard the chloroform extracts. Free the aq. soln. from $CHCl_3$ by centrifuging and pipette 5 ml into a 100-ml calibrated flask. Add 5 ml of N HCl and 5 ml of freshly prepared 0·5 per cent. aq. $NaNO_2$ soln. and mix; after 2 min., add 10 ml of thymol soln. (0·1 per cent. in 1·5 per cent. aq. NaOH) and make up to 100 ml with water. After 2 min., measure the yellow-orange colour in a 0·5- or 1-cm cell with the use of a violet filter. A correction must be applied for unremoved procaine; it is obtained by submitting various concn. (0·01 to 5·0 per cent.) of freshly prepared procaine hydrochloride soln. to the procedure.   H. F. W. KIRKPATRICK

1313. The quantitative estimation of salicylic acid, salicylamide and gentisic acid in the presence of each other in pharmaceutical preparations. R. Rutkowski (*Arzneimittel-Forsch.*, 1954, **4** [3], 209–213).—Four ml of test soln. containing $\ngtr$ 200 μg of Na salicylate are mixed with 1 ml of iron reagent [1·0 g of $Fe(NO_3)_3.9H_2O$ in 500 ml of 1 per cent. aq. HNO_3] and 0·3 ml of Na acetate soln. (8·0 g in 100 ml of water) and the colour is measured after 5 min. Five ml of alcoholic test soln. containing $\ngtr$ 200 μg of salicylamide are mixed with 0·5 ml of iron reagent and the colour is measured after 5 min. In mixtures, the salicylamide is separated by ether extraction and after evaporation of the solvent it is determined as above. Na salicylate may be determined in the presence of Na gentisate by the method given above, as the colour given by gentisate fades rapidly and is very slight after 5 min. A standard curve for gentisate may be used to correct for this absorption. Na gentisate is determined directly in the mixture by adding an excess of 0·1 N iodine to the aq. extract, acidified with acetic acid, allowing to stand for 25 min. and back-titrating with 0·1 N $Na_2S_2O_3$. One ml of iodine $\equiv$ 5·56 $\pm$ 0·11 mg of Na gentisate.   H. F. W. KIRKPATRICK

1314. The quantitative determination of barbituric acid derivatives. H. Bräuniger and G. Borgwardt (*Pharm. Zentralh.*, 1954, **93** [7], 266–269;

[8], 299–303).—Published methods of quantitative determination of barbiturates are reviewed. Results by the mercury perchlorate method of Pedley (*Brit. Abstr. C*, 1950, 370) are good only if the correct initial concn. of barbiturate is taken, although use of aq. methanol as solvent instead of water leads to better results in particular cases under controlled conditions. For general purposes, direct titration in methanol with 0·1 N NaOH (thymolphthalein indicator) is preferred.   A. R. ROGERS

1315. Functional organic analysis. II. Compounds of dimedone (5:5-dimethyl*cyclo*hexane-1:3-dione) with aldehydes. C. Duval and N. D. Xuong (*Anal. Chim. Acta*, 1955, **12** [1], 47–49).—The dimedone derivatives of 26 aldehydes have been prepared and their purity confirmed by i.r. spectrography. Their melting points have been determined (Maquenne block) and their decomp. temp. derived from thermolysis curves.   W. C. JOHNSON

1316. The assay of phenylindanedione. L. K. Sharp (*J. Pharm. Pharmacol.*, 1955, **7** [3], 177–182).—Results of assaying phenylindanedione by a gravimetric technique with dinitrophenylhydrazine were inconsistent. Alkaline solutions of H_2O_2 oxidise the sample to phthalic acid and benzoic acid; since the former is partly destroyed by further oxidation, an arbitrary factor is necessary. Assay of a sample weighing $\ngtr$ approx. 450 mg gave consistent results. Bromination, followed by addition with potassium iodide and titration of the liberated iodine, gives excellent results with 150 to 420-mg samples. The ultra-violet absorption curves of phenylindanedione in various solvents have been obtained, but except when the sample is very small or when foreign matter is present, there is little to recommend spectrophotometric assay. Experimental details of the last three methods are given.   C. H. WHITTON

1317. Determination of methylthiouracil with mercuric chloride. F. Cynajek and J. Szlanga (*Farmacja Polska*, 1954, **10** [1], 17–18; *Referativnȳĭ Zh., Khim.*, 1954, Abstr. No. 29,343).—With Hg^{II} salts, methylthiouracil forms a ppt. of composition $(C_5H_6ON_2S)_2Hg$. A 0·3 to 0·4-g sample of pure methylthiouracil (or methylthiouracil tablets) is dissolved on a water-bath in a mixture of 200 ml of water and 50 ml of a 0·4 per cent. soln. of NaOH. The cooled soln. is made slightly acid to litmus with acetic acid and titrated with 0·05 M $HgCl_2$, or Hg acetate, in the presence of 1 ml of a 0·5 per cent. ethanolic soln. of diphenylcarbazone, until a faint violet colour appears. One ml of 0·05 M $HgCl_2$ is equivalent to 0·01422 g of $C_5H_6ON_2S$. The method is more precise than argentimetric or other methods.   E. HAYES

1318. A method for the determination of 2-carbethoxythio-1-methylglyoxaline. R. A. McAllister (*J. Pharm. Pharmacol.*, 1955, **7** [2], 135–138).—2-Carbethoxythio-1-methylglyoxaline [2-ethoxycarbonylthio-1-methylglyoxaline] (**I**) may be determined by an extension of the method previously described (*Brit. Abstr.*, 1951, 418) for 2-mercapto-1-methylglyoxaline (**II**). *Procedure*—Shake the soln. (3 ml, containing $\simeq$ 100 μg of **I**) gently with N NaOH (1 ml), warm to 37° C for 15 to 20 min., cool, add N HCl (1 ml), followed by borate buffer (pH 8) (5 ml) and 2:6-dichloroquinone-chloroamine (0·1 ml of 0·4 per cent. soln. in aldehyde-free ethanol), stand at room temp. for 20 min., extract with $CHCl_3$ (10 ml), filter the extract through a small filter-paper and measure the optical density

against a similarly treated blank soln. in a Spekker absorptiometer with Chance glass filters (OB1). Calculate by interpolation from a standard curve. **I** gives a negative reaction in the iodobismuthous acid test (*Brit. Abstr. C*, 1952, 397) unless first hydrolysed to **II**.
A. R. ROGERS

1319. The separation of mercury by amalgamation from oxidising solution. A. Berggren and W. Kirsten (*J. Pharm. Pharmacol.*, 1955, **7** [3], 183–184).—The two procedures described for determining mercury in tablets, containing 18·5 mg of 3-chloromercuri-2-methoxypropylurea and other substances (starch, lactose, talc, gelatin, colour) to a total weight of 185 mg, involve amalgamation with Zn in a solution containing free Br. The Br prevents the pptn. of Hg, except on the surface of the zinc, where both Br and Zn are reduced. In the first procedure, 100 ml of water and 0·5 ml of Br were added to a weight of ground tablets containing about 500 mg of 3-chloromercuri-2-methoxypropylurea. After mixing the soln., 10 ml of glacial acetic acid and 1 g of granulated zinc were added. The mixture was first boiled and then digested for 1 hr. After decanting the solution from the amalgam, the latter was well washed with water and dissolved in 10 ml of conc. HNO_3. One per cent. aq. $KMnO_4$ was added until it was no longer decolorised, a further 5 ml were then added. A few drops of 30 per cent. H_2O_2 were added to destroy the permanganate; the solution was then mixed, cooled and titrated with ammonium thiocyanate, ferric ammonium sulphate being used as the indicator. In the second procedure, a weight of powdered tablets corresponding to about 500 mg of pure mercury compound were shaken with 25 ml of H_2O, 0·5 ml of Br and 1 ml of conc. HCl and left to stand for 10 min; 2 g of granulated Zn were added and the mixture shaken for 1 hr. The solution was decanted and the amalgam washed, dissolved and titrated as described in the first procedure. The second procedure gives more accurate and reliable results than the first, in which there may be some loss by volatilisation. Amalgamation without the addition of bromine gave 97 to 99 per cent. recovery with pure 3-chloromercuri-2-methoxypropylurea but only 70 to 80 per cent. with the tablet mixture.
C. H. WHITTON

1320. Alkalimetric assay of sulphates of organic bases in non-aqueous solvents. J.-A. Gautier and F. Pellerin (*Ann. Pharm. Franç.*, 1954, **12** [7–8], 505–508).—Sulphuric acid is too strongly acidic to permit estimation of the sulphates of organic bases by direct titration with perchloric acid in acetic acid. Conductivity experiments show that the sulphate concentration may be sufficiently reduced by the addition of benzidine equivalent to 95 per cent. of the sulphate present, forming the insoluble benzidine sulphate. *Procedure*—To a 0·1 N soln. of the sample in glacial acetic acid, add sufficient benzidine (as 0·05 M soln. in glacial acetic acid) to precipitate 95 per cent. of the sulphate; allow the ppt. to settle, and titrate the supernatant liquid with 0·1 N perchloric acid in glacial acetic acid. The method has been successfully applied to the sulphates of quinine, quinidine, atropine, strychnine, streptomycin, dihydrostreptomycin, framycetin and other organic pharmaceutical bases.
A. R. ROGERS

1321. A study of thermo-electric methods for determining isotonicity. F. M. Goyan and D. Reck (*J. Amer. Pharm. Ass.*, *Sci. Ed.*, 1955, **94** [1], 43–47).—It is proposed that the isotonicity of a given aq. soln. should be expressed as the percentage w/v of a NaCl soln. found by a study of suitable colligative properties to be isotonic with it. Measurements may be made of the slight differences in temp. of micro-samples arising from slight differences in v.p. from that of a selected reference liquid maintained at a constant temp. (such as 30° C). Suitable apparatus and techniques, involving copper - constantan thermocouples and requiring only drops of sample are described.
A. R. ROGERS

1322. On the evaluation of capsicum fruit and tincture of capsicum. G. Schenk (*Dtsch. ApothZtg.*, 1955, **95** [6], 123–124).—Capsicum fruit (of the D.A.B.) should contain ≮ 0·2 per cent. of capsaicin, and tincture of capsicum ≮ 0·02 per cent. Details are given of suitable assays by means of the ammonium vanadate - conc. sulphuric acid reaction. The colour that develops is compared with that produced by a standard soln. of vanillin (*Anal. Abstr.*, 1955, **2**, 453); nine parts of vanillin give the same intensity of colour as one part of capsaicin.
A. R. ROGERS

1323. The application of flame photometry to the assay of some [U.S.] N.F. and U.S.P. solutions. S. W. Goldstein and D. P. Sanders (*Drug Standards*, 1954, **22** [7–8], 137–148).—Practical procedures and techniques are described that utilise the Beckman DU spectrophotometer with flame attachment for the control determination of the cations in pharmaceutical soln. of NaCl, dextrose and NaCl, NaI, KI, potassium citrate, $CaCl_2$, calcium laevulinate and $MgSO_4$. The use of calibration curves or standard solutions is recommended.
A. R. ROGERS

1324. The determination of codeine phosphate in combination with acetylsalicylic acid, phenacetin and caffeine by non-aqueous titration. M. Pernarowski, L. G. Chatten and L. Levi (*J. Amer. Pharm. Ass.*, *Sci. Ed.*, 1954, **43** [12], 746–750).—Codeine phosphate may be determined directly in the presence of acetylsalicylic acid, phenacetin and caffeine, by non-aqueous titration. *Procedure*—Dissolve an amount of sample containing approx. 32 mg of codeine phosphate in phenol (5 g) and $CHCl_3$ (10 ml), filter through cotton-wool, washing with 5 ml of a mixture of phenol (5 g) and $CHCl_3$ (10 ml) for quant. operation; add methyl cyanide (50 ml) and titrate potentiometrically with 0·05 N perchloric acid in dioxan. The mean recovery, based on five samples, is 100·35 per cent., with coeff. of variation 0·64 per cent. The method can be applied to many commercial tablets, but if Mg stearate or gelatin is present as excipient, the following procedure should be employed. Suspend the sample in water (25 ml), add 20 per cent. aq. NaOH (4 ml), extract with $CHCl_3$ (5 × 20 ml), combine the $CHCl_3$ extracts, wash with H_2O (10 ml), filter through cotton-wool, evaporate to 10 ml on a water bath, add methyl cyanide (50 ml) and titrate potentiometrically. The mean recovery for this method is 98·84 ± 0·84 per cent.
A. R. ROGERS

See also Abstracts 1254, 1385, 1415.

Food

1325. Estimation of aluminium in food and biological material. H. Thaler and F. H. Mühlberger (*Z. anal. Chem.*, 1955, **144** [4], 241–256).—The colorimetric determination of Al by means of Eriochrome cyanine R has been investigated and found to give good results with biological material.

Since Cu, Fe and Mn give coloured lakes with Eriochrome cyanine R, they must be removed before estimation of Al. The cupferron method of Meunier can be used, but it is recommended that a pH of 0·4 should be used and not 1·0, since at the latter pH some Al may be precipitated with the Fe. Although Mn does not react with cupferron under the conditions in which Al is precipitated (pH 4·8), some Mn comes down with the Al. Manganous ions do not form a lake with Eriochrome cyanine R and so do not affect the reaction with Al. The small residue of Cu can be reduced with thioglycollic acid and rendered ineffective. Phosphate ions can cause errors in the precipitation of Cu - cupferron. This can be avoided by adding a further quantity of a 5 per cent. solution of cupferron after the separation of the heavy metals at pH 0·4. A cloudiness appears at this pH, owing to the separation of some N-nitrosophenylhydroxylamine. If sufficient of a 20 per cent. solution of ammonium acetate is added to bring the pH up to 4·8 and bring about the precipitation of Al - cupferron, the cloudiness disappears and the aluminium salt is precipitated. Under these conditions 10 to 100 μg of Al can be determined within the normal limits of error in the presence of 100 mg of PO_4'''. F' present in amounts up to 1 mg and SiO_2 from 1·5 to 19 mg are without marked effect, but if larger amounts of SiO_2 are present they must be removed with HF and conc. H_2SO_4. Typical results for the determination of Al in musts and wines and in foods shows that 10 to 100 μg of Al can be determined with an error of 2 to 6 per cent.   A. J. MEE

1326. **Studies on the microscopic and microchemical characterisation of the alkali-metal phosphate particles found in foodstuffs.** C. Griebel (*Z. LebensmittUntersuch.*, 1955, **100** [1], 3–15).—A scheme is given for the characterisation of phosphate particles by microscopy and micro-chemical tests. Microscopic appearance (22 drawings) and reactions with magnesia mixture, $ZnCl_2$, $AgNO_3$, mercuric acetate, hexa-amminocobaltic chloride and o- and p-phenylenediamine hydrochlorides are described for $Na_4P_2O_7$, $Na_2H_2P_2O_7$, $Na_5P_3O_{10}$, $Na_6P_4O_{13}$, $(NaPO_3)_x$, $(KPO_3)_x$ and $(NaPO_3)_3$.   A. R. ROGERS

1327. **Application of complexones to sugar analysis.** M. Potterat and H. Eschmann (*Mitt. Lebensmitt. Hyg., Bern.*, 1954, **45** [4], 312–329).—After a critical examination of the results of sugar determinations with the Luff - Schoorl soln., this soln. has been replaced by one that contains 25 g of $CuSO_4.5H_2O$, 37·22 g of complexone III and 286 g of $Na_2CO_3.10H_2O$ per litre. These compounds are separately dissolved in warm water, the $CuSO_4$ being added last; after being set aside for a few days, the soln. is filtered. The sugar soln. (10 ml, containing 2·5 to 35 mg of sugars) is mixed with 10 ml of the above soln. and refluxed for exactly 10 min. over a naked flame, then rapidly cooled (with the addition of 25 ml of water) and filtered through a sintered-glass filter. The acid soln., obtained by dissolving the Cu_2O from the filter with N HNO$_3$, is approx. neutralised with N aq. NH$_3$, and then made alkaline by the addition of a further 5 to 10 ml. After diluting to 250 ml, and adding murexide indicator, the solution is titrated to a bluish-purple with aq. 0·02 M complexone III. The max. error is $\geqslant$ 0·04 mg of glucose. Further working details and a table of factors for 2·5 to 35 ml of 0·02 M complexone III in terms of mg of invert sugar, lactose and maltose are given.   P. S. ARUP

1328. **Solvent mixtures for chromatographic separation of sugars.** D. B. Das and J. F. Wareham (*J. Sci. Ind. Res., B, India*, 1954, **13** [10], 741–743). —The results obtained with different mixtures of crude benzene and commercial methylated spirits as solvents for the paper-chromatographic separation of sugars are reported. Some results obtained with a (7 + 13) mixture of alcohol and benzene by cellulose-powder chromatography are also given.
                    G. C. JONES

1329. **Determination of fructose in the presence of glucose and other monosaccharides.** D. Karácsony (*Élelmezési Ipar*, 1954, **8**, 309–312).—A new method is described by which 0·2 to 10·0 mg of fructose (or equiv. amounts of fructose-containing sugars, *e.g.*, sucrose) can be determined accurately in the presence of not more than 80 mg of glucose or other aldohexoses, or aldoheptoses, etc. In a 50-ml flask with reflux condenser, put 2 ml of sugar solution, 2 ml of guaiacol solution (20 g in 100 ml of ethanol) and 10 ml of ethanolic HCl (200 ml of conc. HCl to 300 ml of 96 per cent. ethanol). Put the flask in a large bath of boiling water for exactly 10 min., and then in a cold-water bath for 5 min. Pour the solution into a cell and measure the pink colour as soon as possible by means of a photometer with a violet filter. The fructose content can then be read from a previously prepared calibration curve. The pink colour is formed by reaction of the guaiacol with hydroxymethylfurfural formed by degradation of the fructose.   SUGAR IND. ABSTR.

1330. **Studies in barley and malt. I. Effects of water on germination.** R. E. Essery, B. H. Kirsop and J. R. A. Pollock (*J. Inst. Brewing*, 1954, **60** [6], 473–481).—Various germination tests for barley are reviewed. Samples of barley have been allowed to germinate under carefully controlled conditions, with various amounts of available water. The water is shown to be critical. Barleys suitable for malting usually show maximum germination with an excess of water. Barleys of poorer malting quality may show an inhibition with an excess of water. It is suggested that, with these barleys, the process initiating germination takes place slowly and the water penetrates the grain before initiation has taken place, causing an inhibition. However, once the process of initiation has commenced, germination can continue in the presence of excess of water. The preferred method of testing is as follows: 3-cm × 9-cm filter-papers are placed in a 4-in. Petri dish and 4 ml of distilled water are added. Fifty corns are placed in the dish, which is maintained at 18° C. The germinated grains are counted at intervals up to 72 hr. A corn is to be considered germinated when the growing rootlet is clearly discernible or when there is definite evidence of growth of the acrospire. Modifications to malting procedure so that more use may be made of water-sensitive barleys are under consideration.
                    G. B. THACKRAY

1331. **Cereal carbohydrate determination. Sulphonated 1-naphthol and anthrone reactions applied to sulphuric extracts of cereals.** A. W. Devor (*J. Agric. Food Chem.*, 1954, **2** [25], 1290–1292).— Colorimetric methods based on a modified Molisch test and the anthrone test are described for the determination of total carbohydrate and of hexoses and pentoses in the N-free extract of cereals. *Extraction*—The ground material (200 mg) is refluxed for 30 min. with 1·25 per cent. w/v H_2SO_4 (50 ml), the mixture is cooled, diluted to 100 ml with the H_2SO_4, filtered and an aliquot of the

filtrate containing 6 to 8 mg of carbohydrate is diluted to 200 ml with the H_2SO_4. *Determinations*— To 1 ml of sulphonated 1-naphthol reagent (*Anal. Chem.*, 1952, **24**, 1626) and 1 ml of the diluted extract, 5 ml of conc. H_2SO_4 are added rapidly, the mixture is shaken vigorously for 15 sec., heated in boiling water for 8 min. and cooled in ice. The light absorption between 530 and 580 mμ is measured at 10-mμ intervals against a reagent blank, and the amount of carbohydrate present (calculated as monosaccharide, *a* per cent.) is derived by comparison with the light absorption of 1 ml of a standard solution containing 40 μg of glucose treated similarly. With increasing percentages of pentoses in the carbohydrate, the absorption at 580 mμ decreases, while that at 530, 540, 550 and 560 mμ increases. The amount of pentose present is derived by reference to standard curves prepared with the use of increasing percentages of xylose in the glucose standard, while keeping the total carbohydrate at 40 μg. As pentoses give less colour with anthrone reagent than do hexoses, the amount of pentose present can be checked by mixing 5 ml of the diluted extract with 10 ml of anthrone reagent (0·2 g per 100 ml of conc. H_2SO_4), and, after 1 hr. at room temp., measuring the light absorption at 620 and 630 mμ against a reagent blank. The amount of carbohydrate present calculated as monosaccharide (*b* per cent.) is derived by comparison with the light absorption of a standard solution containing 0·20 mg of glucose treated similarly. The approx. percentage of pentose present (calculated as xylose) is given by $115 (a - b) \div a$.
S. C. JOLLY

1332. **Rapid detection of mercury on cereal grains.** D. K. Cunningham and J. A. Anderson (*Cereal Chem.*, 1954, **31** [6], 513–516).—A modification of Lepper's method (*Landw. Vers. St.*, 1933, **117** [1], 109) is described for the detection of normal amounts of common mercury-containing fungicides in small samples of wheat, barley, rye or oats. *Procedure*— Fifteen seeds are boiled for 1 min. in 1 ml of alkaline reducing solution (equal parts of 5 per cent. aq. KOH and 25 per cent. $Na_2S_2O_3$ solutions, freshly mixed) with a piece of aluminium foil (2 mm square $\times$ $^1/_{32}$ in. thick). The foil is removed, rinsed in water, dried with acetone and examined. A positive reaction is shown by small pits on the surface of the foil or by white alumina crystals. The formation of a brick-red stain and bubbles of gas on the foil when it is immersed in 1 drop of 1 per cent. alizarinsulphonate solution in 10 per cent. acetic acid confirms the presence of Hg.
S. C. JOLLY

1333. **Application of amperometric titration to the determination of potassium bromate in flour.** D. K. Cunningham and J. A. Anderson (*Cereal Chem.*, 1954, **31** [6], 517–521).—The accuracy of Armstrong's method (*Brit. Abstr. C*, 1953, 28) for the determination of $KBrO_3$ in flour is improved by an amperometric determination of the iodine end-point. In this simplified procedure, the usual rotating platinum electrode is replaced by a stationary platinum foil (area 2 sq. cm). Allowing for dilution caused by moisture in the flour, the average recovery of $KBrO_3$ is $\simeq$ 97·5 per cent. Standard errors of $\simeq \pm 0·15$ p.p.m. are obtained with flour containing 5 to 15 p.p.m. of BrO_3'; the error is $\simeq \pm 0·37$ p.p.m. at levels of 30 to 40 p.p.m.
S. C. JOLLY

1334. **A method for the rapid determination of moisture in doughs and breads.** H. W. Lincoln,

B. M. Dirks and C. G. Harrel (*Cereal Chem.*, 1954, **31** [6], 506–513).—An infra-red drying method is described by which the moisture content (total volatile matter) of dough and bread can be determined in $\not>$ 10 min.; the results compare well with those given by oven-drying methods. *Procedure*—The sample (5 or 10 g of dough, or 10 g of ground bread) is pressed into a thin layer between two tared Whatman No. 4 filter-papers (18·5 cm) with a laboratory press which develops a pressure of 12,250 lb per sq. in. After separation, the filter-papers are hung, back to back, on the balance arm of a projection-reading balance between two 1550-W infra-red lamps, and dried. The standard error of a single determination was 0·45 per cent. of moisture for doughs and 0·09 per cent. for bread.
S. C. JOLLY

1335. **The determination of lactose in milk altered by lactic fermentation. I. Conditions for quantitative hydrolysis of lactose in the serum.** I. S. L. Ruiz (*Ann. Falsif.*, 1954, **47**, 167–174).—The determination of original lactose in milk that has undergone lactic fermentation is simplified by considering the glucose and galactose formed by hydrolysis as one substance for the purposes of calculation. The simultaneous equations derived from the optical rotation and copper reducing power are thereby simplified and lead to a figure for original lactose that is precise to $\pm$ 1·5 g per litre. Precision for residual lactose is $\pm$ 3 g per litre and for glucose plus galactose $\pm$ 4·5 g per litre. G. B. THACKRAY

1336. **Determination of total acids of ice-cream by luminescence-analysis.** J. Kottász (*Z. LebensmittUntersuch.*, 1955, **100** [1], 54–56).—Lucigenin (dimethyldiacridinium nitrate) shows green chemiluminescence in the presence of H_2O_2 in alkaline soln., and is recommended as an indicator in the determination of total acids of ice-cream. Dyes, sugar, fat and albumin do not interfere, and the results agree within $\pm$ 0·20 per cent. with those obtained when phenolphthalein is used as indicator. *Procedure*—Dissolve the sample (20 ml) in freshly boiled and cooled H_2O (50 to 60 ml), add 3 per cent. aq. H_2O_2 (5 ml), 96 per cent. aq. ethanol (10 ml) and 0·05 per cent. aq. lucigenin (3 to 4 ml); titrate with 0·1 N NaOH in the dark to the first appearance of luminescence. Perform a blank titration, omitting the sample.
A. R. ROGERS

1337. **The detection of preservatives in cheese.** H. Mair-Waldburg and W. Sturm (*Z. Lebensmitt-Untersuch.*, 1955, **100** [1], 51–54).—A scheme of qual. analysis for preservatives in cheese is given. Oxidising agents ($\not<$ 100 p.p.m. of $KBrO_3$ or 1000 p.p.m. of H_2O_2) are detected directly in the cheese, hexamine (50 p.p.m.) is detected in a steam-distillate as formaldehyde, and a toluene extract is tested for salicylic acid (10 p.p.m.), *p*-hydroxybenzoic acid and its esters (50 p.p.m.), benzoic acid (100 p.p.m.) and *p*-chlorobenzoic acid.
A. R. ROGERS

1338. **Studies on the quantitative estimation of egg-yolk in margarine.** E. Becker and W. Clemens (*Z. LebensmittUntersuch.*, 1955, **100** [1], 24–35). —Estimation of egg-yolk in margarine by the determination of lutein, cholesterol or phosphorus is unsatisfactory; the lutein content of egg-yolk is variable, and other constituents of margarine (such as fish oils) may add to the cholesterol or phosphorus content. For water-margarine or milk-margarine of known milk content (when a

correction is applied), good results are obtained by determining the albumin with the aid of ninhydrin. Even if the milk content is unknown, the results are more reliable than those obtained by the lutein method.   A. R. ROGERS

1339. Determination of moisture in chocolate. K. G. Sloman, E. Borker and M. D. Reussner (*J. Agric. Food Chem.*, 1954, **2** [24], 1239).—Water can be determined in chocolate by the Karl Fischer method using the modified reagent suggested by Seaman *et al.* (*Anal. Chem.*, 1949, **21**, 510) standardised with Na tartrate dihydrate. Water is extracted from the sample by heating just to boiling with methanol, the mixture is cooled, reagent A (containing methanol, SO_2 and pyridine) is added and, after 10 min., the mixture is titrated with reagent B (containing iodine and methanol). Low results are obtained when extraction at room temp. with methanol or reagent A is used. Recovery of added water was 90 to 117 per cent. of the total water present and the standard deviation of a single determination is $\pm$ 4 per cent. S. C. JOLLY

1340. A new colour reaction for the determination of glycyrrhizic acid by paper chromatography. J. Gootjes and W. Th. Nauta (*Rec. Trav. Chim. Pays-Bas*, 1954, **73** [11], 886–891).—A standard solution of ammonium glycyrrhizate for the calibration curve, or an extract of liquorice is subjected to paper partition chromatography using as solvent *n*-butanol - ethanol - aq. N NH_3 (60:13:27) giving $R_F \simeq 0.27$. The spot of glycyrrhizic acid is revealed by immersing the paper in a solution of 0.025 per cent. rhodamine 3 GO or rhodamine B in N H_2SO_4 for 15 min. The strips are air dried and exposed to ammonia vapour. The coloured spots are cut out together with a spot of the background of identical size. The colour is dissolved from the paper portions by 20 ml of a mixture of 50 per cent. ethanol and 1 per cent. conc. aq. NH_3, and the difference in extinction is measured with an Engel colorimeter and an S 55 filter.   E. J. H. BIRCH

1341. Malt extract and paper chromatography. M. Lindemann (*Brauwissenschaft*, 1954, **7** [9], 185–189).—Samples of malt and malt liquors were examined qualitatively for their amino-acid and sugar content by paper chromatography, with butanol - acetic acid - water mixtures as solvents. No connection between the amino acids and sugars present during the stages of malting and the solubility of malts could be found, but the degradation of proteins and sugars could be followed during malting.   P. S. STROSS

1342. Further contributions to the forecasting of yield of malt extract from the results of barley analysis. R. Heuss (*Brauwissenschaft*, 1954, **7** [9], 190–196; [10], 223–226).—Eleven formulae for calculating the yield of extract of malt are considered. The general form is Extract = $A - aE + bK$ where A is a constant depending on the type of barley, E is the average weight and K is the average protein content of a corn in g. Results of forecasts made by each are compared with yields of malt extract obtained from the 1952 and 1953 Danish and Bavarian barley crops. The best formulae give forecasts with an error of less than 1 per cent.   P. S. STROSS

1343. Routine determination of carbon dioxide in wine. J. Schneyder and F. Epp (*Mitt. Wein-u. Obstbau, Wien, A*, 1955, **5** [1], 39–42).—The following procedure is carried out with reasonable precautions against undue absorption of atmospheric CO_2. *Procedure*—The sample (10 to 20 ml) is slowly run into 1.5 to 2 times its volume of clear aq. 3.2 per cent. $Ba(OH)_2.8H_2O$ containing 2 ml per litre of 30 per cent. H_2O_2 soln. After boiling and rapidly cooling, the pptd. $BaCO_3$ is filtered (or centrifuged) off, washed with two portions of 10 or 20 ml of water, and transferred with water to a titration flask. After neutralisation to thymol blue with 0.2 N HCl, the mixture is acidified with a known vol. of 0.2 N HCl, and the number of ml of acid used in neutralising the $BaCO_3$ is determined by back-titration with 0.2 N KOH. The conversion factor (ml of acid to g of CO_2 per litre) is 0.44 for 10 ml, or 0.22 for 20 ml of wine. The results agree well with gravimetric determinations.   P. S. ARUP

1344. Determination of the iodine value by the Rosenmund - Kuhnhenn method catalysed with a mercuric salt. A. Doadrio and J. M. Fernández Marzol (*An. Soc. Esp. Fís. Quím., B*, 1954, **50** [12], 981–984).—The use of the Rosenmund - Kuhnhenn reagent without pyridine was confirmed to be satisfactory for determining the iodine values of various oils, and the use of mercuric acetate as a catalyst (*cf.* Norris and Buswell, *Anal. Chem.*, 1943, **15**, 259) gave good results with a contact time of 1 min. The excess of halogen used should be greater than 70 per cent. to avoid low results. The use of the mercuric catalyst method for determining the iodine value of tung oil gives results in accord with theoretical values, but requires at least 2 hr. contact time and the addition of 50 ml of reagent for a sample weighing, *e.g.*, 100 to 140 mg for an iodine value higher than 150. The catalyst should preferably be added before the Br solution.   D. LEIGHTON

1345. Identification of vegetable oils. I. Detection of rape and mustard oils. K. D. Pathak and J. S. Aggarwal (*J. Sci. Ind. Res., B, India*, 1954, **13** [10], 720–722).—Results are reported of investigations carried out to examine the purity of rape and mustard oil samples obtained from different parts of India, and the extent of adulteration in them by other vegetable oils. Determination by Neogi's method (*Analyst*, 1936, **61**, 597) of the lead salt insoluble fraction ("erucic acid number") in the oils was shown to give satisfactory results. The selective oxidation of erucic acid by permanganate oxidation was not satisfactory.   G. C. JONES

1346. Separation of oleic and isooleic acids from hydrogenated vegetable oil by adsorption chromatography. T. J. Boman (*J. Sci. Ind. Res., B, India*, 1954, **13** [10], 718–720).—A method based on adsorption chromatography with alumina as adsorbent and a $(1 + 1)$ benzene - light petroleum (boiling range 40° to 60° C) solvent system has been developed for the separation of oleic and *iso*oleic acids from hydrogenated vegetable oil. The R_F values of oleic and *iso*oleic acids in this solvent mixture were found to be 0.81 and 0.93, respectively. The products separated from hydrogenated vegetable oil have been identified by their melting points. The purity and homogeneity of the acids separated were confirmed by determining their iodine values. Analysis of the products by paper chromatography did not reveal the presence of any short-chain acids.   G. C. JONES

1347. The gravimetric determination of cholesterol in wool wax. H. W. Knol (*Rec. Trav. Chim. Pays-Bas*, 1954, **73** [11], 924–930).—The gravimetric

method (Windaus, *Ber.*, 1909, **42**, 238) for the determination of cholesterol in wool wax as its insoluble addition compound with digitonin is shown to be inaccurate owing to interference from the perhydro*cyclo*pentanophenanthrene alcohols, lanosterol, agnosterol, dihydrolanosterol and dihydroagnosterol. Decomposition and chromatography of the precipitate is used to find its content. It is concluded that the usual colorimetric method is better. E. J. H. BIRCH

1348. Paper chromatography in food analysis. V. Micro-determination of inositol, ethanolamine, serine and choline in phosphatides. H. Sulser (*Mitt. Lebensmitt. Hyg., Bern.*, 1954, **45** [4], 251–294).— The literature of the subject is reviewed. Commercial phosphatides or egg-powder can be prepared for chromatographic analysis, without further purification, by refluxing the sample (30 to 50 mg) with 3 to 4 ml of aq. 6 *N* HCl for 24 hr. in a Gorbach micro-reflux apparatus, filtering the soln. and evaporating to dryness at 70° C and 70 mm of Hg; after taking up the residue in water, filtering and again evaporating to dryness, the residue is dissolved in water up to 200 ml; a suitable aliquot portion (1 to 2 μl) is applied to the starting line of each chromatogram. The following solvents are used: for inositol, ethyl acetate - acetic acid - water (3:1:3); for ethanolamine and serine (on the same chromatogram), *n*-butanol - ethanol - water (80:25·5:60); and for choline, *tert*.-butanol - ethanol - water (4:5:1); the spraying reagents used are, respectively, methanolic AgNO₃ (2·5 per cent.), followed by exposure to NH₃ vapour, ethanolic ninhydrin (0·2 per cent.) and (for choline) a 1 per cent. soln. of molybdophosphoric acid in CHCl₃ - ethanol (1:1) followed by 1 per cent. SnCl₂ in 3 *N* HCl. Further working details are given. The chromatograms are evaluated by the line-spectrum photometric method of Sulser and Högl (*cf. Brit. Abstr. C*, 1953, 365). Examples are given of chromatograms from egg and rape phosphatides, in which several unknown substances have been detected. Determinations of choline in egg-powder agree well with determinations made gravimetrically by the reineckate method. P. S. ARUP

1349. Rapid assay for vitamin A. N. Scott (*Chemist Analyst*, 1954, **43** [4], 97).—The following procedure requires only one transfer, no special glass and only a small vol. of ether. *Procedure*—A sample containing ≃ 100 U.S.P. units of vitamin A is weighed into a conical centrifuge tube, 0·35 ml of ethanolic KOH (50 per cent.) and 5 ml of ethanol are added, and a cork and air-condenser are attached. After refluxing for 15 min. on a water-bath, 5 ml of H₂O and 10 ml of ether are added, and the tube is stoppered and shaken for 3 min. The tube is centrifuged and all but a drop of the aq. layer removed with a syringe. Five ml of H₂O are added, and the tube is shaken and centrifuged and the aq. layer is removed as before. Two more washings with H₂O are given; the ether is evaporated with N through a syringe, and 10 ml of *iso*propanol are added. The optical densities at 310, 325 and 334 mμ are compared with those of a blank solution that has undergone the above operations, and the Morton - Stubbs correction is applied. H. P. PAGET

1350. Paper chromatography of vitamin K₁ and related compounds with some observations on products of ultra-violet irradiation. J. P. Green

and H. Dam (*Acta Chem. Scand.*, 1954, **8** [8] 1341–1346).—Reversed-phase paper chromatography is applicable to the separation and identification of naphthaquinones. Their R_F values were inversely related to the length of the side-chain in the 3-position up to a 30-carbon grouping. The sensitivity of the method was 0·5 μg for qual. and 1·5 μg for quant. analysis. The most useful solvent system is *iso*propanol - acetic acid - water (60:2·5:37·5 by vol.). Exposure of the spots to a u.v. lamp for 45 to 60 sec. induced a permanent change in the fluorescent colour of most compounds. Spraying with ethanolic KOH after activation in this way produced yet other colours which were stable for at least four months. Among the compounds examined were menadione, phthiocol and vitamins K₁ and K₂. N. E.

See also Abstract 1275.

Sanitation

1351. Water examinations by membrane-filter and most-probable-number procedures. P. Kabler (*Amer. J. Publ. Hlth.*, 1954, **44**, 379–386).—A study has been made by the Standard Methods Committee for the Examination of Water and Sewage to compare the membrane-filter technique and the standard fermentation tube method of evaluating the most-probable-number of bacteria for the routine examination of water. There was no apparent relation between the agreement or disagreement of results by the two methods and any recorded physical or chemical characteristic of the water samples, except that the turbidity limits the volume of sample which can be examined by the molecular filter method. It appeared that the two methods do not measure exactly the same group of organisms. Of 1706 samples examined, 74 per cent. of the results obtained by the two methods were in agreement; if differences were due to sampling only, an agreement of 90 to 95 per cent. would be expected. Further studies are necessary to determine the sanitary significance of the differences in the results. WATER POLLUTION ABSTR.

1352. Determination of sulphate ion in natural water by use of ion-exchange resin and disodium ethylenediaminetetra-acetate. K. Isagai (*J. Chem. Soc. Japan*, 1954, **75** [6], 613–616).—The sample is passed through a column of cation-exchange resin (Amberlite IR-120) (1·5 drops per sec.) to remove Ca·· and Mg··. A known excess of BaCl₂ is added to the filtrate to ppt. BaSO₄ and the excess of Ba·· is titrated with disodium ethylenediamine-tetra-acetate standard soln. with Eriochrome black T as indicator. A small known amount of Mg·· is added to make the end-point more clear, since Mg·· gives a more stable coloured compound with the indicator than Ba··. This method is simpler than the method of Munger *et al.* (*Brit. Abstr. C*, 1951, 127) for the determination of SO₄″ in natural water. K. SAITO

1353. Determination of silica in water by co-precipitation with aluminium hydroxide. M. Akahane and C. Tanaka (*J. Chem. Soc. Japan*, 1954, **75** [2], 140–143).—For the determination of silica in water, neither the gravimetric method nor the colorimetric method is satisfactory; the former involves a tedious evaporation and the latter does not determine colloidal silica. Silica contained in water is co-pptd. quant. with ≃ 15 times its weight of alumina at pH 8·5 ± 0·2. The ppt. is dissolved

in 2 N HCl and Si is determined either gravimetrically ($SiO_2 > 1$ mg per litre) or colorimetrically ($SiO_2 < 1$ mg per litre). When the amount of Si is < 1 mg per litre, a blank test should be carried out. K. SAITO

1354. Review of methods for cyanide determination in sewage. D. F. Krawczyk (*Sewage Ind. Wastes*, 1954, **26** [11], 1388–1392).—The general methods in use for cyanide determinations in sewage have been reviewed and some have been adopted by the Buffalo Sewer Authority after suitable amendment to permit the determination of small amounts. By each of the three methods, namely, the Liebig titration method, the benzidine - pyridine method and the pyridine - pyrazolone method, a blank solution is obtained by destruction of the cyanide present by treatment of the filtered sewage with Cl and NaOH at pH 10. This solution and another portion of the filtered sewage are treated with the appropriate reagent for colorimetric determination. A. WEBSTER

1355. Methodological studies on "square-diluent" method for testing disinfectants. R. L. Stedman, E. Kravitz and H. Bell (*Soap, N.Y.*, 1954, **30** [11], 132–133, 137, 139 and 152).—The development of a new performance test procedure termed the "square-diluent" method is summarised. The method consists of inoculating one-inch squares of surface material (stainless steel, linoleum, etc.) with a mixture of *Micrococcus pyogenes* var. *aureus* cells, *Salmonella schottmuelleri* cells and *Trichophyton interdigitale* spores with or without serum, drying the inoculum, adding disinfectant to the dried films, permitting reaction for 10 min. and recovering the survivors by plating in different media. Calculations of the percentage reduction are made. G. HELMS

1356. The isolation of γ-hexachlorocyclohexane from oily solution for purposes of chemical identification. W. Hoffmann and E. Hoffmann (*Arzneimittel.-Forsch.*, 1954, **4** [10], 631–633).—Two methods are described for the isolation of Gammexane in a pure form from oily soln. The first is an extraction method in which the Gammexane is removed by shaking out with acetic anhydride. After separation of the anhydride, it is mixed with an equal vol. of ethanol or methanol and evaporated to a small vol.; more alcohol is added, the evaporation is continued and the soln. is allowed finally to crystallise in the cold. The crystals are collected and recrystallised from ethanol; after drying, the m.p. is 112° to 112·5° C. In the second method, the oily soln. is distilled with half its wt. of phenylacetic acid at a temp. of 270° to 275° C on using a fractionating column warmed with another burner. The distillate is dissolved in boiling water, cooled and treated with $NaHCO_3$; when all of the CO_2 has been evolved the soln. is shaken with ether. The ether is treated with fresh $NaHCO_3$, dried with $CaCl_2$ and evaporated. The residue is recrystallised from ethanol, dried and the m.p. taken. The first method gives the better results. H. F. W. KIRKPATRICK

See also Abstract 1249.

Agriculture and Plant Biochemistry

1357. The analysis of emulsifiable parathion preparations. L. Westenberg (*Meded. Landb-Hoogesch., Wageningen*, 1954, **19** [3], 554–557).—The colorimetric determination of parathion (**I**) in

technical preparations can be carried out, after removal of free nitrophenol, with the use of a strongly basic ion-exchange column. *Procedure*—Dissolve or homogenise a quantity of the preparation, equivalent to 40 mg of **I**, in 96 per cent. ethanol and dilute to 50 ml. To an aliquot (12 ml), add 96 per cent. ethanol (58 ml) and water (100 ml), cool and dilute to 200 ml. Pass the solution, in 35 per cent. v/v ethanol, through an ion-exchange column containing De-Acidite F or Amberlite IR-4B in the hydroxide form, reject the first 170-ml fraction and collect the next 25 ml. To this fraction, add NaOH (0·5 g), set it aside at room temperature for 24 hr. and measure the absorption in a 4- or 5-mm cell, using a blue filter (Jena BG 12). Since results depend on dimensions of the column and speed of passage, some trials should be carried out with pure (non-emulsifying) **I** solutions. P. S. STROSS

1358. Detection of O-(3-chloro-4-nitrophenyl)-OO-dimethyl phosphorothioate [chlorthion] and analysis of residues in milk. M. J. Kolbezen and J. H. Barkley (*J. Agric. Food Chem.*, 1954, **2** [25], 1278–1280).—With minor modifications, the method of Averell and Norris (*Brit. Abstr. C*, 1949, 170) for the determination of parathion is applicable to the determination of chlorthion. In preparing the standard curve, the reduction period with Zn is increased to 30 min. and the final coloured solution contains slightly more acid (6 ml of conc. HCl per 100 ml). For the determination of chlorthion residues in milk, two alternative extraction processes are described; one is essentially the method of Dahm *et al.* (*J. Dairy Sci.*, 1950, **33**, 747) and the other is a more rapid method that requires mixing in a Waring Blendor for only 1 min. The extracted material is reduced with Zn and a procedure is described for removing the milk fat and other interfering substances before developing the colour. Concentrations of $> 0·1$ p.p.m. in 500 ml of milk can be determined spectrophotometrically, and 0·02 to 0·1 p.p.m. can be estimated by visual comparison in Nessler tubes. Beer's law is obeyed with 20 to 300 μg of chlorthion per 100 ml and the absorption max. is at 545 mμ. S. C. JOLLY

1359. Colorimetric determination of OO-dialkyl 1-hydroxyphosphonates derived from chloral. P. A. Giang, W. F. Barthel and S. A. Hall (*J. Agric. Food Chem.*, 1954, **2** [25], 1281–1284).—A colorimetric method, based on the pyridine - alkali reaction with $CHCl_3$, described by Fujiwara (*Sitzber. naturforsch. Ges. Rostock*, 1916, **6**, 33), is described for the determination of a new series of organic phosphorus insecticides prepared by condensing chloral with a dialkyl phosphite. The method was developed by using OO-dimethyl 2:2:2-trichloro-1-hydroxyethylphosphonate (Bayer L 13/59). *Procedure*—The sample (25 to 300 μg) is heated for 1 hr. at $\simeq$ 550° C in a micro-furnace and the pyrolysis products are drawn steadily through 6 ml of a pyridine - water mixture (50 ml of water and 400 ml of redistilled colourless pyridine) in an absorption tube that is cooled in an ice - salt mixture ($-15°$ to $-20°$ C). A further 3 ml of the pyridine - water mixture are added to the absorption tube followed by 1 ml of 0·25 N NaOH, the tube is loosely stoppered, heated for 3 min. in boiling water and cooled immediately in running water. The solution is filtered through paper, and the colour, which is stable for 3 min., is measured in a colorimeter with a green filter (500 to 570 mμ). The amount of ester is derived by reference to a standard curve prepared from

pure ester (recryst. from light petroleum containing a little benzene; the dibutyl ester is purified by repeatedly washing a benzene solution with water until free from chloral). The water solubilities of the diethyl, dipropyl and dibutyl esters have been determined using this method, which is sensitive to 20 μg.   S. C. JOLLY

1360. Insecticide residue analysis. Sodium reduction technique for micro-determination of chlorine in organic insecticides. W. F. Phillips and M. E. DeBenedictis (*J. Agric. Food Chem.*, 1954, **2** [24], 1226–1228).—A modified sodium reduction method is described for the routine determination of residues of organic chlorine insecticides in food products with an accuracy comparable to that of combustion techniques. A 1 to 2-kg sample is extracted for 5 min. with 500 ml of Skellysolve B, the solvent is decanted, shaken with 20 g of anhydrous Na_2SO_4 and 4 g of Filter Cel and filtered, and the volume is noted. The extract is passed through a bed consisting of a mixture of $CaCO_3$ and Celite No. 545 (1 + 1), the bed is washed with 200 ml of Skellysolve B, and the combined eluate and washings are evaporated to dryness. The residue is refluxed for 60 min. with 10 ml of *iso*-butanol and 1 $\pm$ 0·2 g of Na and, when cool, the excess of Na is destroyed by refluxing for 5 min. with 10 ml of aq. *iso*propanol (1 + 1 by vol.). After cooling to about 16° C, 5 ml of acetone are added, the solution is exactly neutralised to phenolphthalein with dil. HNO_3 (1 + 1 by vol.), transferred to a 50-ml flask and successively rinsed in with 2 ml of the HNO_3, 4 ml of 1 per cent. gelatin solution and sufficient aq. acetone (1 + 1 by vol.) to dilute to 50 ml. A 10-ml aliquot is titrated amperometrically with standard $AgNO_3$ solution at the rotating platinum electrode. The strength of the $AgNO_3$ solution affects the accuracy of the method; for solutions containing 0·1 to 10·0 μg of Cl per ml, approx. 0·001 N $AgNO_3$ should be used; for concn. of 10 to 100 μg of Cl per ml, 0·01 N $AgNO_3$ is recommended. For standardising the $AgNO_3$ solution, 5 reagent blanks are prepared by refluxing 1 g of Na with 10 ml of *iso*butanol and diluting to 50 ml as described before. Two sets of 10-ml aliquots of each of the 5 solutions are titrated with 0·01 and 0·001 N $AgNO_3$ (average titre for each set = reagent blank). To 3 sets of 9-ml aliquots of each of the 5 solutions, 1 ml of a standard NaCl solution is added to give concn. of 0·1, 0·5 and 1·0 μg of Cl per ml for the respective sets, the solution is titrated with 0·001 N $AgNO_3$, and the normality calculated from the set showing the highest accuracy and least standard deviation; for 0·01 N $AgNO_3$, one set of aliquots containing 0·5 μg of Cl per ml is used. The titres should not exceed 3 ml.   S. C. JOLLY

1361. A note on polarography and chromatography of some commercially available chlorophyllins. W. L. Wuggatzer and J. E. Christian (*J. Amer. Pharm. Ass., Sci. Ed.*, 1955, **94** [1], 30–31).—Ten commercial chlorophyllins have been studied polarographically in addition to those of the previous report (*Science*, 1953, **118**, 444). In 0·1 M KCl, all show a reduction wave with $E_{\frac{1}{2}} = -1·20$ to $-1·38$ V *vs.* the S.C.E. In 1·5 M aq. NH_3 - NH_4Cl, six show a reduction wave with $E_{\frac{1}{2}} = -0·87$ to $-1·02$ V; the other four are insol. in this medium. The fractions responsible for the wave were separated by adsorption chromatography on talc, H_2O and 0·1 M KCl being used as developer. In most cases, the $E_{\frac{1}{2}}$ values of the separated fractions were at more negative potentials than those of the original preparations.   A. R. ROGERS

1362. Canavanine: detection and occurrence in Colutea arborescens. W. R. Fearon and E. A. Bell (*Biochem. J.*, 1955, **59** [2], 221–224).—Guanidinoxy compounds, including canavanine (α-amino-δ-guanidinoxybutyric acid) give a purple colour with aq. sodium nitroprusside at pH 5 to 7·5, whereas alkylguanidines, including arginine, react only at pH values greater than 8. Determination of guanidinoxy compounds is carried out as follows: to the neutral solution (1 ml) (containing 0·1 to 1·0 mg of canavanine), add 1 per cent. aq. sodium nitroprusside (0·5 ml). Dilute to 10 ml with phosphate buffer (pH 7), mix and expose to daylight for 40 min. A standard solution is treated similarly. Compare the optical densities of the two solutions by means of a visual or photo-electric colorimeter with a blue or blue-green filter. The amount of canavanine is ascertained from a standard graph. The method detects canavanine in concn. as low as 0·008 per cent.   J. N. ASHLEY

1363. The analysis of triterpenes. I. Colorimetric estimation of the triterpene acid content in the Labiatae. C. H. Brieskorn and M. Briner (*Arch. Pharm., Berlin*, 1954, **287** [8], 429–432).—Treatment of ursolic or oleanolic acids with HSO_3Cl gives a violet colour and subsequent treatment (with strong cooling) dropwise with H_2O (avoiding carbonisation) affords a greenish product, which on $CHCl_3$ extraction to exhaustion gives a yellow extract, that in a photo-electric colorimeter shows colour proportional to the original quantity of material. For the determination, the accurately weighed drug is first extracted with light petroleum (boiling range 45° to 60° C) and then repeatedly with boiling methanol, the extract being made up to volume, halved and evaporated to dryness in separate flasks. One half, on treatment with HSO_3Cl and decomposition with H_2O as above gives, on $CHCl_3$ extraction, a soln. whose optical density, after subtracting the blank value of the untreated half, gives a triterpene acid content (from a standard graph) in good agreement with results obtained gravimetrically.
                                F. R. MUMFORD

1364. Determination of fusaric acid by paper chromatography. H. Zähner (*Phytopath. Z.*, 1954, **22** [2], 227–228).—The acid is adsorbed from the culture-filtrate on to Norit-A powder (2 g per 100 ml), from which it is eluted by a mixture of methanol (100 ml) and 25 per cent. aq. NH_3 (10 ml). The eluate is concentrated by evaporation under reduced pressure. Isolation of the acid (R_F 0·83 to 0·85) is effected by chromatography, 0·1 ml of the concentrate and, as ascending solvent, butanol - formic acid - water (75 : 15 : 10) being used. After spraying with aq. bromocresol green (reacting in the alkaline range), the fusaric acid spot (pink under yellow) is excised and eluted with ethanol at 90° C during 3 hr. The fusaric acid is determined spectrophotometrically in the eluate at 268 mμ (not at 227 mμ) in comparison with a corresponding eluate obtained by starting with a known vol. of aq. 1 per cent. synthetic fusaric acid. A blank test is conducted with the eluate from an excised piece of the chromatogram paper of the same area as that used for the tests. The paper shows a strong absorption at 227 mμ.   P. S. ARUP

1365. Polarographic behaviour of juglone; determination of juglone in the presence of ascorbic acid in the fruits and green parts of the walnut (Juglans regia). P. Zuman (*Chem. Listy*, 1954, **48** [4], 524–532).—The polarographic behaviour of juglone (**I**) in the absence of boric acid resembles

that of other naphthaquinones. A reversible polarographic wave is preceded by an adsorption wave. From the dependence of the $E_\frac{1}{2}$ of $\mathbf{I}$ on the pH, the dissociation exponents were determined, pK_{r_1}, 7·0; pK_{ox}, 8·7; pK_{r_2}, $\simeq$ 11·0. In the presence of boric acid at pH > 6, complex formation occurs, manifested by the formation of a new wave and by an increased stability towards alkaline hydrolysis.

G. GLASER

1366. Further constituents of hawthorn. Paper-chromatographic identification of chlorogenic acid and caffeic acid. U. Fiedler (*Arzneimittel-Forsch.*, 1954, **4** [1], 41–45).—A description is given of the paper chromatography of chlorogenic acid and caffeic acid prepared from coffee beans by the use of various solvents. The extraction and identification of these substances in the leaves, fruit and flowers of the hawthorn (*Crataegus*) is also described.

H. F. W. KIRKPATRICK

1367. Observations on nucleic acid and polyphosphate in *Torulopsis utilis*. R. Chayen, S. Chayen and E. R. Roberts (*Biochim. Biophys. Acta*, 1955, **16** [1], 117–126).—Investigation of the nucleic acid fraction of yeast obtained by the Schneider technique (*J. Biol. Chem.*, 1945, **161**, 293) has revealed the presence of an additional phosphorus compound; it is suggested that this is a linear polyphosphate of high mol. wt. Because of this, a modified method for the detection of P in the Schneider fraction has been developed that permits the determination of nucleic acid phosphate and polyphosphate in the presence of each other. The method depends on a differential hydrolysis in 5 per cent. trichloroacetic acid for 1 hr. at 90° C.

G. W. CAMBRIDGE

1368. Spectrochemical analysis. I. Spectrochemical determination of trace metals in synthetic plant-ash materials. J. M. Gillette (*Dissert. Abstr.*, 1954, **14** [10], 518).—A method is proposed for analysing the trace elements in maize leaves, viz., Fe, Cu, Mn, Co, Mo and B. The material is dry-ashed and the residue is taken up in HCl solution. Constant amounts of Sn (internal standard), Mg and K_2SO_4 (spectrochemical buffers) are added. Standard solutions are prepared for comparison. A drop of the test or standard solution is evaporated to dryness on a $\frac{1}{4}$-in. graphite rod with a slight cavity in the end. The residue is burned in a 5-amp. 4 kV a.c. arc. The spectra are observed through a large Littrow quartz spectrograph and recorded on Kodak Spectrum Analysis No. 1 plates. Standard methods of photometry are used to construct working curves; a seven-step rotating sector intensity pattern is placed on each plate for emulsion calibration. The accuracy is from 1 to 10 per cent.

S.C.I. ABSTR.

1369. Studies on methods of determination of trace elements in the living body. V. Determination of [zinc in] plant ash by polarography. I. S. Hakomori and K. Yamamoto (*J. Chem. Soc. Japan*, 1954, **75** [2], 132–135).—The sample (100 to 150 g of the air-dried plant) is charred at 200° to 300° C in a platinum dish and ashed below 600° C (4 to 5 hr.). A 1-g portion of the ash is dissolved in 6 N HCl, filtered and made up to 100 ml. A known vol. is taken and the pH is adjusted to 3·0 to 4·0 with aq. NH_3; the polarogram is plotted and the Zn content is calculated.

K. SAITO

1370. Relationships of humic and fulvic acids in various types of soil. S. Cecconi and M. Tellini (*Ann. Chim., Roma*, 1954, **44** [11], 943–948).—

Spectrophotometric determination of the ratio of the optical densities of extracts of soils with 0·1 M $Na_4P_2O_7$ solution at 470 mμ and 690 mμ offers a rapid method for discovering the predominance of complex molecules (humic acids), or of comparatively simple ones (fulvic acids). If the ratio $E_{470m\mu}$ to $E_{690m\mu}$ is < 5, humic acids are the main component; if > 6·5, then fulvic acids predominate. Greater accuracy is attained by carbon analysis before and after acidification with H_2SO_4 at pH 2 and separation of the pptd. humic acids.

C. A. FINCH

See also Abstract 1154.

5.—GENERAL TECHNIQUE AND LABORATORY APPARATUS

General

1371. Aids to semi-micro qualitative analysis. D. A. Pantony and C. L. Wilson (*J. Chem. Educ.*, 1954, **31** [12], 648).—Details are given for the construction of several pieces of apparatus designed to facilitate semi-micro quantitative analysis. These include modified analysis boards, hanging-drop cells and H_2S-precipitation cells. A method of suspending semi-micro test-tubes in a water bath is also described.

A. LEDWITH

1372. A micro gas generator. L. C. W. Baker and J. E. Stouffer (*J. Chem. Educ.*, 1954, **31** [11], 593).—Details are given for the construction and operation of a small Kipp generator for H_2S. The instrument uses the conventional reagents (FeS and HCl), but eliminates fume and generator problems. With slight modification, the instrument can be used for CO_2 and other gases.

A. LEDWITH

1373. Simple controllable air leak for a vacuum system. D. C. McDonald (*N.Z. J. Sci. Technol., B*, 1954, **36** [3], 207).—The arms of a stopcock are removed and the holes sealed. It is then inserted into a rubber bung closing an inverted test-tube, which is connected at the other end to the apparatus. When the pressure in the apparatus is reduced sufficiently the plug of the stopcock "floats," allowing a constant current of air to enter. The air leak is controlled by weights placed on a balance pan suspended from the plug.

A. M. SPRATT

1374. New filter-flask. M. Potterat and H. Eschmann (*Mitt. Lebensmitt. Hyg., Bern*, 1954, **45** [4], 329–331).—A shortened Allihn sintered-glass filter-tube is fused into the side of a round 100-ml flask to make an angle of 120° with the neck of the flask. In the boiling position, the neck of the flask, which carries a reflux condenser, is inclined at 45°; in the filtering position, the inside of the flask can easily be washed by a jet of water. The apparatus is convenient for sugar determinations.

P. S. ARUP

1375. Modified Pregl hot micro-filtration apparatus. V. Horák (*Chem. Listy*, 1954, **48** [4], 616).—A modified design of the Pregl micro-filtration apparatus is described, in which the filter stick connected by means of a ground-glass joint to an external jacket is heated by the vapours of the solvent.

G. GLASER

1376. A variable flow device for accurately delivering small flows of liquids. D. E. Weiss (*Chem. & Ind.*, 1954, [52], 1573).—A constant stream of drops of liquid is obtained by lowering a rod steadily

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into a vessel filled with the liquid to the height of an overflow tube. The rod is lowered from the driving chain of a clock movement driven by a weight, and the rate of lowering is altered by adjusting the length of the pendulum.

A. M. SPRATT

1377. A laboratory apparatus for charging and discharging pipettes and the like. G. F. Persson and K. V. Persson (Brit. Pat. 720,204, Date Appl. 19.3.52).—An apparatus for charging and discharging pipettes is described. It embodies a device for holding the pipette in jaws actuated with a lever against a resilient pad that has a hole leading to a rubber bulb, and carrying different forms of valve (which are described) for inter-connecting the pipette and the bulb. An auxiliary fine-adjustment bulb is also described. E. J. H. BIRCH

1378. A new combustion furnace for elementary analysis. R. Grewe (Z. anal. Chem., 1955, **144** [5], 321–322).—A furnace is described that accommodates twin combustion tubes made of porcelain, and stationary burners, which heat the whole combustion tube in stages. Diaphragm pumps giving a constant gas flow are used to provide the current of CO_2 or N. The absorption tubes are shaped so that they can easily be handled with tongs and fit on any micro-balance. P. S. STROSS

1379. Electrometric Karl Fischer titration. J. G. van Pelt and H. Keuker (Chem. Weekbl., 1955, **51** [7], 97–99).—A simple apparatus for carrying out the Karl Fischer titration electrometrically is described. A. J. MEE

1380. Measurement of very small amounts of moisture in gases. A. Perlick and R. Perlick (Kältetechnik, 1954, **6** [10], 271–274).—The friction coeff. (F) between carbon brushes and a rotating metal rod depends on the formation of an adsorbed layer on the carbon surface, which depends on the nature of the ambient gas and the partial pressure (p) of water vapour therein. A copper rod inside a glass tube having two side tubes containing the spring-loaded brushes is rotated by a 1-watt d.c. shunt-wound motor; gases to be tested are led through the tube after the speed has been adjusted by a resistance in series with the armature winding, the two being in parallel with the respective ratio arms of a bridge. A change in the motor speed causes, across the bridge, an unbalance e.m.f. (e), which over a small range is proportional to the speed change and hence to F. For nitrogen, e falls as p rises. Argon, C_2H_2 and CO_2 show similar effects, but these are small for CO_2. For air and oxygen, e and p increase together, probably because the graphite particles worn away are oxidised. In general, the greater the heat of adsorption of a gas the higher is F. The method is applicable to 0·05 to 5 mg of H_2O per cu. metre. It can be applied to organic vapours. A. R. PEARSON

1381. Humidity recorder for gases. L. von Ripka (Chem.-Ing.-Tech., 1954, **26** [8–9], 440).—The gas passes through a chamber containing a thin metal mirror, which reflects a convergent beam of light on to a photo-cell if the temp. of the mirror is above the dew-point of the gas. The mirror forms one wall of a cell through which flows gas or liquid at a temp. controlled by current amplified from the photo-cell. A thermocouple is soldered to the back of the mirror and connected to a millivoltmeter, the pointer of which is a registering pen actuated by current from the photo-cell. The temp. of the mirror oscillates

about the dew-point of the gas, which is indicated and periodically recorded at 30- to 60-sec. intervals.

A. R. PEARSON

1382. A new gas-sampling apparatus. W. R. Dudden (Lab. Practice, 1955, **4** [2], 71–72).—In a new gas-sampling apparatus described and illustrated, the plug of the bottom tap of the sampling bulb has one or more hemispherical depressions. As the plug is rotated, the depression contacts the gas stream, fills with gas and is carried round until it contacts mercury in the sampling bulb. This gas is then displaced with mercury and a bubble of gas rises through the mercury into the top of the sampling bulb, whilst on further rotation of the plug, the Hg falls from the depression and is discharged through the seal, being replaced by a further sample of gas. Sampling can be automatically controlled by specific-gravity changes, from flame characteristics, by clock control or by pressure control. C. H. WHITTON

1383. A development of gas analysis by the Orsat apparatus. W. Hunsmann (Chem.-Ing.-Tech., 1954, **26** [8–9], 437–439).—Hydrocarbons (C_2 to C_4) are condensed in two bulbs that are connected in series between the sampling burette and the reagent pipettes and cooled by liquid nitrogen or solid carbon dioxide. For gas mixtures containing $\simeq$ 50 per cent. of C_3 to C_4 hydrocarbons, suitable temp. for the bulbs are $-90°$ C and $-145°$ C. The uncondensed constituents pass on, swept by a measured vol. of N, into a second burette and thence to the usual reagent pipettes and combustion tube containing CuO. The condensates are then in turn vaporised into the burette and similarly analysed. Results on known mixtures of paraffins and olefins having more than four carbon atoms show an accuracy equal to that of the normal Orsat analysis. A. R. PEARSON

1384. Azotometers. Imperial Chemical Industries Ltd. (Inventor: F. R. Cropper) (Brit. Pat. 720,882, Date Appl. 15.4.53).—The tap at the top of an azotometer on which the grease is easily attacked by strong alkali is replaced by a capillary tube leading down after two right-angle bends to a tap, above which is a mark on the tube. During the collection of gas in the azotometer, mercury from a reservoir is kept level with this mark. The gas is removed from the azotometer by opening the tap and allowing the mercury to fall below the level of a side opening, by which the gas may leave. The nitrogen in the capillary is not removed and the alkali does not come into contact with the tap.

E. J. H. BIRCH

1385. Simplification of the apparatus for estimation of acid hydrazides by gas analysis. C. M. P. Wirth (Dtsch. ApothZtg., 1954, **94** [52], 1289).—A simplification is described of the apparatus of Harting (Brit. Abstr. C, 1953, 396) for the determination of acid hydrazides (e.g., isonicotinic hydrazide) by oxidation to N with potassium ferricyanide and measurement of the vol. of N formed. The simplification consists of a wide-necked 250-ml bottle (**I**) fitted with a double-bored rubber bung. One bore is fitted with a tap and the other connects by means of rubber tube to the top end of a 25-ml measuring burette of a nitrometer containing water. Procedure—The acid hydrazide (50 to 100 mg in 20 ml of water) is introduced into **I** and 10 ml of 20 per cent. NaOH solution are added. Into a glass or plastic 25-ml container standing upright in **I**, 10 ml of a 20 per cent. solution of $K_3Fe(CN)_6$ are

introduced from a pipette. The bung is then inserted into **I** with the tap open. After standing for 10 min., the tap is closed and the water in the burette brought to the zero mark. The two solutions in **I** and the container are then brought into contact, shaken for two min. and, after standing for 15 min., the vol. of N produced is measured. The amount of hydrazide is calculated from the following equation

$$R \cdot CO \cdot NH \cdot NH_2 + O \rightarrow R \cdot CHO + H_2O + N_2.$$

H. L. WHITEHEAD
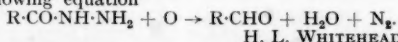
1386. Reduced-scale Reid vapour-pressure apparatus. R. L. LeTourneau, J. F. Johnson and W. H. Ellis (*Anal. Chem.*, 1955, **27** [1], 142–144).—An apparatus for measuring the Reid vapour pressure of only $\simeq$ 2 to 3 ml of volatile non-viscous petroleum products consists of a sample cup, to fix the liquid - vapour ratio (generally $\simeq$ 1:4), tightly clamped to a pressure-transducer which converts the pressure into an exact electrical equivalent that is measured differentially by a detector circuit. Corrections for change in air pressure on heating and for change in v.p. of water are eliminated by heating the vapour space to 100° F before commencing the test. The pressure range is 0 to 20 lb. and the whole procedure occupies about one-third less time than the standard methods. Full operating details are given. W. J. BAKER

1387. A rapid-acting semi-micro vapour-density apparatus. J. T. Stock and M. A. Fill (*J. Chem. Educ.*, 1954, **31** [12], 656).—Details are given for the construction and use of a rapid-acting semi-micro vapour-density apparatus of the Victor Meyer type. Two types of vaporising chamber are described and experimental results are given for both types when chloroform was used as the test substance. A. LEDWITH

1388. An automatic separating column for analytical use. G. Gottschalk (*Z. anal. Chem.*, 1955, **144** [5], 342–347).—An ion-exchange column arrangement is described and illustrated, suitable for use with all ion-exchange resins. It is designed for work in either an upward or downward direction and this accelerates separations as well as keeping the volume of eluate to a minimum. P. S. STROSS

1389. A rotational viscometer for surface films. S. C. Ellis, A. F. Lanham and K. G. A. Pankhurst (*J. Sci. Instrum.*, 1955, **32** [2], 70–73).—A ring of platinum wire coated with paraffin wax and connected by a torsion wire to a synchronous motor is floated on a film-covered surface. The viscous drag by the film on the ring is calculated from the angular displacement between the ends of the torsion wire. G. SKIRROW

1390. An automatic-recording analytical balance. E. J. Caule and G. McCully (*Canad. J. Technol.*, 1955, **33** [1], 1–11).—The design, construction and operation of an automatic-recording balance unit is described. The controller-recorder may be attached to an analytical balance. No mechanical changes in the balance are necessary and there are no connections to the beam or spring. The design embodies no precision fitting but is built up of standard parts and a few parts that can be made in the laboratory. The restoring force for unbalances in weight is the force between a primary coil and a short-circuited secondary coil. The sensing element for unbalance consists of the same two coils, use being made of the dependence on distance apart of their mutual inductances. The short-circuited coil hangs from the balance. I. JONES

1391. Some sensitive and recording volumeters. B. Kok (*Biochim. Biophys. Acta*, 1955, **16** [1], 35–44).—Kinetic measurements in photosynthetic problems require volumeters of accuracy and speed beyond that of the standard equipment. The theoretical considerations involved in the design of such equipment are considered and details of the construction of several types of volumeters of high absolute and relative sensitivity are given. A servo-system for recording gaseous exchange in such volumeters is described; the full scale deflection represents 0·01 μl or more as required. This may be applied to any apparatus in which a fluid meniscus serves as a null detector. The sensitivity of the gasometric apparatus was not affected by this recording unit. G. W. CAMBRIDGE

1392. Simple apparatus for comparing emulsions and suspensions. D. A. Pearce (*Anal. Chem.*, 1955, **27** [1], 163–164).—The emulsion- (or suspension-) comparator consists of a wood frame in which are fitted six glass cylinders 10 in. high by 1½ in. in internal diam. Each cylinder is calibrated by means of a 20-cm mark (corresponding to volumes of 185 to 195 ml) and the emulsions are prepared in the cylinders, the frame being usually rotated by hand. Procedures for the direct comparison of conc. and dil. emulsions, wettable spray powders (for suspension and foaming), etc., are described. Unless introduction of the emulsion concentrate into the water is almost simultaneous with stirring, variable results are obtained for emulsions containing < 1 per cent. of oil. W. J. BAKER

1393. Automatic counting and size analysis of microscopic particles. H. Nassenstein (*Chem.-Ing.-Tech.*, 1954, **26** [12], 661–667).—This review covers the automatic counting and grading of particles in aerosols by light scattering and contact electric impulse methods, and on microscope slides by the double scan, intercept length, guard spot and memory techniques; indirect, direct and semi-direct methods are used, and special mention is made of the flying spot microscope. Limits of size, accuracy and counting rate are discussed. A. B. DENSHAM

1394. Portable electrostatic dust sampler with electronic air flow. D. G. Beadle, P. H. Kitto and P. J. Blignaut (*Arch. Ind. Hyg.*, 1954, **10** [5], 381–389).—An electrostatic dust sampler, suitable for use in gold mines that have a very low airborne-dust content, is described. The air is ionised (positively, to reduce ozone formation) by passing it between wires (diam. 0·006 in.) carrying an ionising current of 400 to 500 μA at 12,000 V. The parallel collecting plates for the charged dust particles have a p.d. of 6000 V. In the absence of draught, the apparatus generates an "electric wind" of $\not<$ 40 cu. ft. per min., and high collecting efficiency is maintained with speeds of > 100 cu. ft. per min. for 12 to 15 hr. The collecting cell (wt. 12 lb) can be operated by a separate power unit with small 6-V batteries (total wt. is then 42 lb) or from the mains supply. C. E. SEARLE

See also Abstracts 1120, 1287.

Optical

1395. A simple ratio-recording spectrometer. N. H. E. Ahlers and H. P. Freedman (*J. Sci. Instrum.*, 1955, **32** [2], 61–64).—A double-beam attachment for a standard single-beam infra-red spectrometer is described. A single radiation beam from a globar source travels alternately through

the control and sample cells, which are arranged as a rotating sector assembly in front of the entrance slit of the monochromator.   G. SKIRROW

1396. An evacuable die for the pressed potassium bromide technique.   H. Hausdorff (*Applied Spectroscopy*, 1954, **8** [3], 131–136).—A die chamber in the form of a split cone facilitates removal of the disc after pressing.   B. S. COOPER

1397. Logarithmic sector *vs.* photo-electric densitometer in measuring intensity ratios.   M. Green and M. L. Polk (*Applied Spectroscopy*, 1954, **8** [3], 126–130).—A simple attachment is described for use with a comparator for density matching of wedged lines obtained with a logarithmic sector. This method of estimating relative intensities, although adequate for many problems, is less accurate than the conventional photo-electric densitometer method. With a group of lines in the hydrogen spectrum in the neighbourhood of 4100 A, the logarithmic sector method gave a cofficient of variation of about 4 per cent., whereas photo-electric densitometry gave 2 per cent.
B. S. COOPER

1398. Fully automatic equipment for colorimetric analysis.   H. Fuhrmann (*Chem.-Ing.-Tech.*, 1954, **26** [7], 401–404).—For the completely automatic colorimetric analysis of solutions and gases in works processes, discontinuous or continuous methods of measurement can be used. Apparatus is described, which is based on these principles, for the analysis of coloured solutions and gases. In the continuous methods, reagents are added automatically; in the continuous coloration is measured photometrically.
R. J. MAGEE

1399. [An improved filter for] flame photometers.   Evans Electroselenium, Ltd. and G. C. Collins (Brit. Pat. 712,700, Date Appl. 10.9.51).—A burner is supplied with a mixture of combustible gas, air and the test solution, which is atomised through a stainless-steel fine-capillary tube. The light from the burner flame is passed through a filter, which filters out all the light except that of the colour produced in a colourless flame by the burning of the element to be determined. This light is then measured to determine the quantity of the sample element, *e.g.*, K in a mixture of K and Na salts, by a photo-electric cell and a galvanometer.
J. M. JACOBS

1400. [Objective lens for] electron microscopes.   Philips Electrical Industries, Ltd. (Brit. Pat. 711,672, Date Appl. 12.6.52; Neth. Pat. 15.6.51).—The magnetomotive force in the lens is such that the lower limit of magnetic saturation is reached in the pole-pieces, which have bores of diameters $\not> \frac{1}{3}$ of the distance between the pole faces. The space between the pole faces is long enough for a focus to be located therein, but as short as is compatible with the insertion of an object between the focus and that pole-piece which is traversed first by the electrons.   J. M. JACOBS

See also Abstract 1184.

Thermal

1401. Simple device for unattended maintenance of "dry ice" temperature.   A. E. O'Keeffe, J. C. Holmes and F. A. Morrell (*Rev. Sci. Instrum.*, 1954, **25** [12], 1225).—A thermally insulated box containing 10 lb of "dry ice" is provided with a heat-conducting rod projecting from the inside of the box to an external cold-trap.   G. SKIRROW

1402. A simple electric heater for inflammable liquids.   S. M. Charlett (*Lab. Practice*, 1955, **4** [2], 70–71).—A simple inexpensive heater for inflammable liquids is described and illustrated. An electric-light bulb is used as the heating element. Tests showing the heating requirements of various liquids and the fairly wide range of control are reported. The liquid being heated is illuminated at the same time.   C. H. WHITTON

1403. Electric heater for van Slyke - Folch carbon combustion apparatus.   L. V. Hankes (*Anal. Chem.*, 1955, **27** [1], 166–167).—Illustrated details are given of the design and construction of a small electric heater (110 W, 115 V) for the easy regulation of heat and temp. during a van Slyke - Folch manometric carbon combustion by the wet method (*Brit. Abstr. A II*, 1941, 24). The heater, which replaces the original micro gas-burner, is controlled by a variable auto-transformer (or a rheostat for d.c.) mounted in a small aluminium box, which is electrically insulated, where necessary, by plastics cemented to the walls.   W. J. BAKER

1404. Water-flow safety switch for gas- or electric-heaters.   R. P. Harpur (*Anal. Chem.*, 1955, **27** [1], 168).—A simple device is shown for preventing failure of water-pressure during cooling of the condenser in a mercury diffusion-pump. A lever-arm micro-switch is mounted above a glass tube (20 cm by 3·5 cm), which serves as a float-chamber, containing a cork float connected electrically to the switch. The return water from the condenser enters the float-chamber and leaves at the base at a controlled rate below that needed for cooling the system. An increase in rate of flow raises the float to switch on the heater, whilst a decrease switches off the heater. The heater does not function initially until the water has been turned on. A magnetic gas-valve is necessary for adapting the above safety-switch to a gas-burner.
W. J. BAKER

1405. A calorimeter for determining specific heats of liquids.   R. G. Charles (*J. Chem. Educ.*, 1954, **31** [11], 577).—A calorimeter is described in which a chemical reaction is used as the source of heat. Constructional details are given for the instrument, which is made almost entirely of glass. Heat is generated by neutralising standard aq. H_2SO_4 with a soln. of standard NaOH. The instrument has been used mainly at room temperature and the results are reproducible to about $\pm$ 0·02 cal. per g per °C. The experimental results show an average deviation of $\pm$ 0·01 cal. per g per °C from the values given in International Critical Tables.
A. LEDWITH

1406. The accuracy of moving-bomb calorimetry. II. Equilibration of carbon dioxide between the gaseous and the liquid phase in the bomb.   L. Bjellerup and S. Sunner (*Rec. Trav. Chim. Pays-Bas*, 1954, **73** [11], 862–870).—The conditions of rotation of the bomb, that are necessary for equilibrium between gaseous and liquid phases in rotating-bomb calorimetry for compounds containing C, H, O and halogen or C, H, O and S, are investigated.
G. SKIRROW

1407. A simple melting-point apparatus.   A. Gero (*J. Chem. Educ.*, 1954, **31** [12], 645).—Details are given for the construction of a simple melting-point apparatus. The design ensures that the thermometer and sample are evenly heated and melting points of excellent reproducibility are obtained.
A. LEDWITH

1408. A new melting-point apparatus. T. Singh (*J. Chem. Educ.*, 1954, **31** [12], 646).—The construction of a new type of melting-point apparatus is described. The apparatus is designed to eliminate a slight temperature difference that has been observed between the centre and the outside edge of the silicone bath in the modified Hershberg melting-point apparatus. A. LEDWITH

1409. Dew-point measuring device. Illinois Testing Laboratories, Inc. (Brit. Pat. 711,584, Date Appl. 11.7.52, U.S. Pat. 13.7.51).—The device is based on the fact that at the instant that the first molecules of dew are deposited the vapour will commence to give up its heat of vaporisation, so tending to raise the temp. of the moisture-collecting "mirror" and change its rate of cooling. This change of rate is detected by incorporating a thermocouple in the moisture-detecting device. J. M. JACOBS

1410. Temperature of contraction of fibres as an aid to identification. Apparatus for test. J. N. Banks and E. R. Johnston (*J. Text. Inst.*, 1954, **45** [12], T941–T943).—A simple electrically heated device for measuring the m.p. of thermoplastic fibres to within 1° C is described. (See Abstract 1411 below.) L. VALENTINE

1411. Temperature of contraction of fibres as an aid to identification. Reply. J. M. Preston and A. S. Brown (*J. Text. Inst.*, 1945, **45** [12], T943).—It is pointed out that the m.p. of thermoplastic fibres depends on the conditions, *e.g.*, presence of oxygen and rate of heating, so that it is more convenient to quote a melting range than a m.p. for identification purposes. (See preceding abstract.) L. VALENTINE

Electrical

1412. Electromagnetic laboratory valve. B. P. McKay and C. H. Eades, jun. (*Anal. Chem.*, 1955, **27** [1], 163).—An electromagnetic valve for controlling the flow of alkali to an automatic-recording titrator (*Anal. Abstr.*, 1955, **2**, 1415) is described. The valve, which is operated by a 40-V a.c. supplied to the 115-V solenoid, is automatically opened and closed by a line-operated pH-sensitive controller, and can be used continuously with no significant rise in temp. The valve is greaseless, easily cleaned, and fast-acting. W. J. BAKER

1413. Self-balancing system for continuous control of current or voltage. F. J. Dunn, J. B. Mann and J. R. Mosley (*Anal. Chem.*, 1955, **27** [1], 167–168).—Analysis of binary gas mixtures, *e.g.*, of isotopes, by the thermal conductivity method consists in passing a constant current through a wire in the gas and measuring the wire's resistance. A circuit and closed-loop servo-system is described for automatically controlling the apparatus during a continuous series of isotope separations. The critical factor affecting sensitivity is the heating current (0·5 amp.), normally supplied by three thermostatically controlled 120-amp.-hr., 12-V cells in parallel; a galvanometer sensitive to 0·02 μV per mm is included. By adding a 5-ohm resistor in series with the conductivity bridge and a 500-ohm 10-turn servo-driven Helipot plus a 200-ohm fixed resistor in parallel with the first, the current can be controlled to 1 in 10^5; a reproducibility of 0·02 per cent. is given. A servo-circuit for continuous control of voltage (to $\simeq$ 10 μV) of an electric furnace is also shown. W. J. BAKER

1414. Versatile polarographic cell. R. L. Pecsok and R. S. Juvet, jun. (*Anal. Chem.*, 1955, **27** [1], 165–166).—A robustly constructed cell for analysing strongly acidic or basic solutions (2 to 55 ml) which, in the normal dilution cells, slowly dissolve the agar plug, is described and sketched. The arrangement prevents diffusion of Cl′ from the reference electrode into the test solution. A glass electrode can be permanently mounted in the stopper if necessary. W. J. BAKER

1415. Automatic titrating and recording apparatus for microbiological assays. C. H. Eades, jun., B. P. McKay, W. E. Romans and G. P. Ruffin (*Anal. Chem.*, 1955, **27** [1], 123–127).—The instrument described and illustrated can automatically titrate and record results on 225 microbiological samples consecutively with a standard error of < 0·5 to 1 per cent. and an accuracy of more than 97 per cent. The apparatus consists of a pH-sensitive electronic controller, a glass-calomel electrode assembly, a sample-carrier and automatic sample-changer, a solenoid-operated polystyrene valve, an automatic recording internal-timer and electronic manometers for maintaining const. pressure of nitrogen on the alkali used for titration and on the stirring device. The whole assembly can be modified for application to assays of vitamins and amino acids, acid-base titrations, oxidation-reduction titrations, and similar procedures. Full constructional and operational details are given. W. J. BAKER

1416. Some aspects of the oxygen electrode. H. R. Gersmann (*Chem. Weekbl.*, 1955, **51** [8], 109–114).—A review of the work on the oxygen electrode is given. The theory of the electrode is discussed with special reference to the adsorption theory, the hydrogen peroxide theory and the oxide theory. None of the theories so far advanced explains satisfactorily all the observed facts, but that of Berl, requiring the formation of hydrogen peroxide, seems to be the best for alkaline media. The experimental work of Hoar is reviewed, but it is doubtful whether the explanation of his results is of general application. It is concluded that the oxygen electrode is useful empirically for potentiometric titrations and the determination of pH.

A. J. MEE

1417. Metallic electrodes for pH measurement. I. J. Jackson (*Lab. Practice*, 1954, **3** [11], 454–458).—Electrodes are divided into equilibrium electrodes (including the hydrogen, quinhydrone and air electrodes) and corrosion electrodes (antimony and tungsten electrodes). Their properties and uses are reviewed. G. SKIRROW

1418. The use of chromium metal as indicator electrode in electrometric titrations. A. Riad Tourky, I. M. Issa and H. Khalifa (*Anal. Chim. Acta*, 1954, **11** [6], 563–568).—A chromium electrode can be used as an indicator electrode in potentiometric acid-alkali and redox titrations. Between pH 3 and pH 10, the response of the electrode corresponds to 48·5 mV per pH unit at 30° C and it is therefore unsuitable for the determination of pH values, unless previously calibrated. pK_a values calculated from titration curves of the weaker acids are not in good agreement with accepted values. The results of a number of redox titrations indicate that the electrode can be used in alkaline or fairly strong acid solutions and in solutions of strong reducing agents. The end-point inflexions are generally smaller than those obtained with the platinum electrode. W. C. JOHNSON

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1419. Contribution to the method of paper electrophoresis. M. Eggstein and H. Hundeshagen (*Klin. Wochschr.*, 1955, **33** [1–2], 14–15).—The design of an apparatus for the separation of proteins in 0·005 to 0·01-ml amounts of serum is illustrated. The conditions for the separation of lipo- and glycoprotein in 0·1 to 0·2 ml of serum are reported.
G. W. CAMBRIDGE

1420. A new apparatus for automatic coloration and extraction of the filter-paper strips in electrophoresis on filter-paper. H. K. Oosterhuis, G. Prins and H. Verleur (*Rec. Trav. Chim. Pays-Bas*, 1954, **73** [11], 963–968).—An automatic apparatus for the extraction of surplus dye from a number of paper strips, simultaneously, is described. The extraction bath is fed with tap-water to which 1 per cent. of acetic acid is added by an automatic device, and the bath is automatically emptied and refilled by relays when the temp. reaches 65° C.
E. J. H. BIRCH

1421. Continuous electrophoresis on paper. G. Dicastro and M. San Marco (*J. Chem. Soc.*, 1954, 4157–4159).—A new apparatus for continuous paper electrophoresis is described. A high degree of uniformity of the electric field, and constancy of pH and ionic concn. are maintained over the whole of the sheet.
A. JOBLING

1422. A photo-tube circuit with transistors. J. P. Phillips and R. W. Keown (*J. Chem. Educ.*, 1954, **31** [11], 605).—Details are given for the construction of a unit to eliminate or avoid the circuit complications arising from the use of vacuum photo-tubes in colorimeters. The unit is cheap to construct and a colorimeter containing such a unit is superior in sensitivity to the barrier layer cell circuits commonly available.
A. LEDWITH

1423. Improved mass-spectrometer ion source for the analysis of solids. J. D. Louw and J. W. L. DeVilliers (*Rev. Sci. Instrum.*, 1954, **25** [12], 1226–1227).—Design and constructional details are given.
G. SKIRROW

1424. Trap for attenuating mercury vapours in the mass spectrometer. B. L. Tuffly and W. J. Lambdin (*Anal. Chem.*, 1955, **27** [1], 164–165).—Passage of mercury vapour (mainly from the sample-inlet) into the ionisation chamber of a mass spectrometer can be stopped by inserting a trap filled with 10-mesh zinc (A.R. grade) followed by a liquid-oxygen trap between the mercury orifice and the metal valve-block; ball-joints facilitate removal of the zinc-trap for cleaning and refilling. Diffusion of zinc is prevented by fitting platinum gauze across the two openings; background effects from absorbed materials are minimised by heating the trap with resistance-wire (50° to 100° C). The trap, which needs changing only after six months' continuous use, can be used when organic compounds are being examined. About two weeks after installing the attenuator, samples admitted through the mercury orifice show strongly decreased peak heights at m/e values of 101.
W. J. BAKER

1425. Mass-spectrometer analyses using a viscous leak. H. C. Mattraw, R. E. Patterson and C. F. Pachucki (*Applied Spectroscopy*, 1954, **8** [3], 117–120).—Use of a mass spectrometer for plant-stream analyses involves the reduction of the higher pressures of the plant stream to the low operating pressure necessary for the mass spectrometer. The capillary-tube leak (viscous leak) is a suitable device for the purpose, but dimensional factors (diameter, length and constriction) affect the calibration. Even for multi-component mixtures, gas sensitivities are a function of the square root of the average molecular weight of the mixture, hence the need for preparing standard mixtures of composition approximately that of the analytical sample is no longer necessary.
B. S. COOPER

Translations

The following papers of interest to analytical chemists have been translated into English. Copies of these papers can be obtained from Consultants Bureau, 152, West 42nd Street, New York 18, N.Y., U.S.A. Each translation costs $7.50 and orders should state title, author(s) and English page number. The English page number is given in parentheses after the Russian page number.

These translations can also be seen in the library of the Chemical Society, Burlington House, London, W.1.

J. Appl. Chem., U.S.S.R.—

Light filters for colorimetric analysis in the ultra-violet region of the spectrum—S. N. Andreev and R. I. Gindina, 1952, [1], 77 (65).

Determination of small quantities of carbon disulphide and acetone by ultra-violet colorimetry—S. N. Andreev and R. I. Gindina, 1952, [1], 104 (89).

Effect of silicic acid on the determination of aluminium in solutions using the *ortho*hydroxyquinoline method—S. K. Dubrovo, 1952, [11], 1151 (1213).

Determination of copper in the pigment monastral blue—N. P. Kanyaev and A. A. Spryskov, 1952, [11], 1220 (1277).

A rapid industrial control method for determining reducing sugars—B. O. Lyubin, 1953, [5], 556 (517).

The quantitative and qualitative determination of acetone and acetophenone in the presence of ethyl methyl ketone—V. I. Tikhonova, 1953, [6], 662 (621).

An apparatus without rubber connections for distillation of hydrochloric acid—V. A. Aleksandrov, 1953, [10], 1097 (1035).

A new method for the quantitative determination of lignin—K. Kürschner and T. Shveitspakherova, 1953, [11], 1176 (1115).

Determination of lignin by the method of Kürschner and Shveitspakherova—K. Kürschner and T. Shveitspakherova, 1953, [11], 1186 (1125).

Potentiometric titration of divalent iron, divalent platinum and tervalent iridium when present simultaneously—A. A. Grinberg, N. B. Karpenko and E. A. Maksimyuk, 1953, [11], 1107 (1047).

Bull. Acad. Sci., U.S.S.R.—

Conditions for the separation and gravimetric determination of silicic acid. II. Polymerisation of silicic acid—E. N. Egorova, 1954, [1–2], 16 (13).

J. Anal. Chem., U.S.S.R.—

English translations of all papers published in *Zh. Anal. Khim.*, *SSSR*, between January, 1953, and June, 1954, are also available.

ABBREVIATIONS

Certain abbreviations in everyday use are not included in the following list. When any doubt might arise from the use in the text of an abbreviation or symbol the word is printed in full.

alternating current	a.c.	millicurie	mC
ampere	amp.	milligram	mg
Ångstrom unit	A	millilitre	ml
anhydrous	anhyd.	millimetre	mm
approximate, -ly	approx.	millimicron	$m\mu$
aqueous	aq.	millivolt	mV
atmospher-e, -ic	atm.	minimum	min.
boiling-point	b.p.	minute (time)	min.
British thermal unit	B.Th.U.	molar (concentration)	M
calorie (large)	kg-cal.	molecul -e, -ar	mol.
calorie (small)	g-cal.	normal (concentration)	N
centimetre	cm	number	no.
coefficient	coeff.	observed	(obs.)
concentrated	conc.	ounce	oz
concentration	concn.	part	pt.
critical	crit.	patent	pat.
crystalline	} cryst.	parts per million	p.p.m.
crystallised	} cryst.	per cent. wt. in wt.	per cent. w/w
cubic	cu.	per cent. wt. in vol.	per cent. w/v
current density	c.d.	per cent. vol. in vol.	per cent. v/v
cycles per second	c.p.s.	potential difference	p.d.
decompos -ing, -ition	(decomp.)	pound	lb
density	ρ	precipitate	ppt.
density, relative	d or wt. per ml	precipitated	pptd.
derivative	deriv.	precipitating	pptg.
dilute	dil.	precipitation	pptn.
direct current	d.c.	preparation	prep.
distilled	dist.	qualitative, -ly	qual.
electromotive force	e.m.f.	quantitative, -ly	quant.
electron-volt	eV	recrystallised	recryst.
equivalent	equiv.	refractive index	n
experiment	expt.	relative humidity	R.H.
foot, feet	ft.	revolutions per minute	r.p.m.
gram	g	saponification value	sap. val.
gram-molecule	mole	saturated calomel electrode	S.C.E.
half-wave potential	$E_{\frac{1}{2}}$	second (time)	sec.
horse-power	h.p.	soluble	sol.
hour	hr.	solution	soln.
hydrogen ion concentration	[H·]	specific gravity	sp. gr.
hydrogen ion exponent	pH	specific rotation	$[\alpha]$
inch	in.	square centimetre	sq. cm
infra-red	i.r.	standard temperature and	
insoluble	insol.	pressure	s.t.p.
kilogram	kg	temperature	temp.
kilovolt	kV	ultra-violet	u.v.
kilowatt	kW	vapour density	v.d.
maxim -um, -a	max.	vapour pressure	v.p.
melting-point	m.p.	volt	V
microcurie	μC	volume	vol.
microgram	μg	watt	W
microlitre	μl	wavelength	λ
micron	μ	weight	wt.
milliampere	mA		

In addition the following symbols are used—

greater than	$>$	less than	$<$
not greater than	$\not>$	not less than	$\not<$
is proportional to	$\propto$	of the order of, approximately	$\simeq$

The principal Pharmacopoeias are denoted by B.P., U.S.P., or D.A.B., together with the identifying numeral.

Radicles are represented by the usual symbols; positive ions have superscript dots and negative ions superscript dashes, *e.g.*, Cu··, Al···, Cl′, SO₄″. Metals that exist in more than one valency state are represented by their symbols with appropriate superscript roman numerals, *e.g.*, ferric iron becomes Fe[III] and cuprous copper Cu[I].

ANALYTICAL ABSTRACTS

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